

4.2 *Pressure Reducer* — An accessory required for counters operated at atmospheric pressure, it should preferably use expansion of the gas through a critical orifice.

5 Test Method

NOTE 2: The details of sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice.

5.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing air, nitrogen or argon, believed to be free of particles of 0.1 micrometers or more in diameter, through the instrument and recording the total number of counts. A suggested assembly for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. A method recommended by the instrument manufacturer, such as internal recirculation through a filter, may be substituted for the system shown. Count a minimum of 8 sample intervals, each at least 25 standard liters (0.95 SCF) or 30 minutes, whichever is greater. Calculate \bar{X}_B as defined in Section 3. \bar{X}_B must not exceed 2 particles per 25 standard liters.

5.2 The sampling point should be at outlet of system, and sampling lines should be as short as possible.

5.3 A suggested sampling probe configuration for turbulent main line flow is shown in Figure 2. The flow rate in the sampling tube at pipeline pressure should be set so that the mean sampling flow velocity at the probe inlet matches as closely as possible the axial flow velocity in the pipeline. The pitot sampling tube ID should be no less than 2 mm (0.08 inch). The orifice and sampling horn should be sized so that the mean flow velocity at the particle counter probe inlet matches the axial flow velocity in the horn as closely as possible.

5.4 Count the particles in each of at least 8 sample intervals. Each sample interval must be at least 25 standard liters or 30 minutes, whichever is greater. Record the number of counts and the sample volume for each interval. Calculate \bar{X}_C and S_C , as defined in Section 3.

6 Specification

6.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 4.

6.2 The specification will be considered met if the calculated concentration of particles plus two standard

deviations does not exceed 10 particles per 25 standard liters, i.e.:

$$\bar{X}_C + 2 * S_C \leq 10 \text{ particles/25 standard liters}$$

7 Report

7.1 The report shall contain the values of all the variables defined in Section 3.

8 Precision

8.1 This test procedure defines the requirements to satisfy the specification at the 95% confidence level.

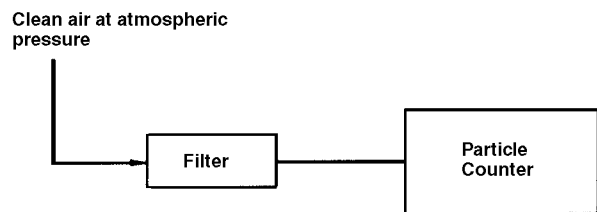


Figure 1
Suggested Assembly for Determining Particle Counter Background

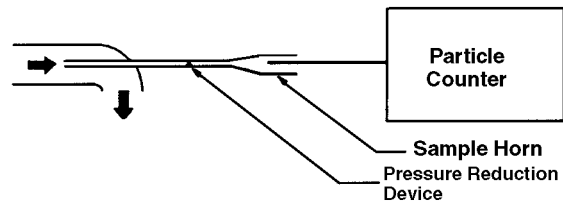


Figure 2
Schematic Diagram of Configuration for Obtaining Particle Samples from Pipelines

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SEMI C6.7-93 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 NITROGEN IN HIGH PRESSURE GAS CYLINDERS

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 1993.

1 Purpose

1.1 The purposes of this document are (1) to set a maximum permissible particle concentration for 10/0.2 grade cylinder nitrogen, and (2) to describe a reference method for its verification.

2 Scope

2.1 This specification applies to nitrogen contained in high pressure gas cylinders; it is not applicable to pipeline gases.

2.2 A nitrogen cylinder consists of three components: (1) a cylinder bottle, (2) compressed nitrogen contained within, and (3) a cylinder valve. Each component can be a particle source. This specification applied to the total number of particles detected in the gas as obtained from the cylinder under a prescribed condition. No consideration is given to the origin of the particles.

2.3 It is known that pressure reduction, if not controlled, can produce a large number of artifact particles through nucleation and condensation. To avoid this complication, this specification adopts particle counters that can be operated at a pressure up to 200 bar (3000 psi), eliminating the need for pressure reduction and its associated problems.

2.4 It is known that particle content in cylinder gases varies with time because particles can be lost to cylinder walls by diffusion or sedimentation, and detached from cylinder walls by flow pulses or mechanical shocks. It is important to measure particle concentration under the worst conditions which represent typical handling of gas cylinders. This specification describes a standard shock test that fulfills the above requirements and provides a procedure to count particles immediately after the shock.

2.5 If this test method is to be used for more than one cylinder, then each cylinder must be tested. It is known that particle contamination in cylinder gases is a strong function of the handling history of the individual cylinders. Cylinders in the same batch of filling are usually returned from various customers after various periods of service. Uniform quality can not be assumed for the same batch of cylinders unless each of them has gone through a dedicated process that erases the

memory of previous history prior to filling. Therefore, batch sampling at 10% or 20% cannot be accepted because of the significant differences among cylinders.

2.6 The requirement of 100% sampling restricts the total amount of gas in each cylinder that can be used for sampling purposes. This restriction, in turn, calls for certain relaxation of the statistical requirement for particle sampling at low concentration levels.

2.7 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 Terminology

3.1 Variables

X_{Bi}	Observed particle concentration in the i^{th} sample interval of the background
X_{Mi}	Observed particle concentration in the i^{th} sample interval before shocks
X_{Ni}	Observed particle concentration in the i^{th} sample after shocks
N_B	Number of sample intervals of the background
N_M	Number of sample intervals before shocks
N_N	Number of sample intervals after shocks
\bar{X}_B	Averaged observed particle concentration of the background
\bar{X}_M	Averaged observed particle concentration before shocks
\bar{X}_N	Averaged observed particle concentration after shocks
X_P	Calculated particle concentration before shocks
X_Q	Calculated particle concentration after shocks
S_B	Standard deviation of \bar{X}_B
S_M	Standard deviation of \bar{X}_M
S_N	Standard deviation of \bar{X}_N
SE_P	Standard deviation of X_P
SE_Q	Standard deviation of X_Q

3.2 Averaged Observed Concentration (X_B , X_M , X_N)

$$\bar{X}_B = \text{SUM}(X_{Bi})/N_B$$

$$\bar{X}_M = \text{SUM}(X_{Mi})/N_M$$

$$\bar{X}_N = \text{SUM}(X_{Ni})/N_N$$

3.3 Calculate concentration (X_P , X_Q)

$$X_P = \bar{X}_M - \bar{X}_B$$

$$X_Q = \bar{X}_N - \bar{X}_B$$

3.4 Standard Deviation (S_B , S_M , S_N)

$$S_B = [\text{SUM}(X_{Bi} - \bar{X}_B)^2 / (N_B - 1)]^{1/2}$$

$$S_M = [\text{SUM}(X_{Mi} - \bar{X}_M)^2 / (N_M - 1)]^{1/2}$$

$$S_N = [\text{SUM}(X_{Ni} - \bar{X}_N)^2 / (N_N - 1)]^{1/2}$$

3.5 Standard Error (SE_P , SE_Q)

$$SE_P = [(S_M^2/N_M) + (S_B^2/N_B)]^{1/2}$$

$$SE_Q = [(S_N^2/N_N) + (S_B^2/N_B)]^{1/2}$$

4 Apparatus

4.1 High-Pressure Particle Counter (HPC) — An instrument capable of counting particles in compressed gaseous nitrogen at a pressure up to 200 bar (3000 psi), having a minimum counting efficiency of 50% at 0.2 μm and reaching 100% at 0.25 μm . This value is determined at ambient pressure by the instrument manufacturer using 0.2 μm monodisperse particles and a reference counter with a proven counting efficiency of 100% at 0.2 μm . Note that the nominal flow rate of the HPC is fixed at any pressure but the sample flow rate should be adjusted to within 5% of the manufacturer's specified flow rate, for the pressure and reported its equivalent standard flow rate at ambient pressure (1 bar).

4.2 Impact Shock Device — A device that can reproducibly impart an impact shock of 10^4 m/sec^2 with a 10% tolerance to a gas cylinder. A convenient set up as shown in Figure 1 can be used. A steel ball is attached to a chain with the other end fastened to the test cylinder. The ball is lifted to form a 90° angle with the cylinder, is released to follow a 90° free fall arc, and strikes the test cylinder. The desired ball mass and chain length are to be determined by monitoring the corresponding shock intensity by an accelerometer. For a typical 44 liter cylinder, a ball mass of 160 gm and a chain length of 60 cm can produce the desired impact shock.

5 Test Method

5.1 Determine the averaged background concentration (\bar{X}_B) by passing particle-free, compressed nitrogen through the HPC. Count a minimum of 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time period of 6 minutes, whichever is greater. A high purity gas filter can be used to remove particles greater than 0.2 μm . Calculate \bar{X}_B as defined in Section 3. \bar{X}_B must not exceed 1 particle per 30 second liters.

5.2 Set up the experimental apparatus as shown in Figure 2. Directly connect the test cylinder to a High-pressure Particle Counter (HPC), a pressure gauge, and a Flow Control Device (FCD). Note that NO REGULATOR is used before the HPC. The sampling line should be clean and as short as possible. The flow control device can be a metering valve and a flowmeter or a critical orifice.

5.3 Determine \bar{X}_M by opening the cylinder valve and count the particles for at least 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time of 6 minutes, whichever is greater. Calculate X_P and SE_P as defined in Section 3.

5.4 Apply 4 impact shocks of approximately 10^4 m/sec^2 each, 10 sec apart, to the bare surface of the test cylinder. Each shock should be applied at the approximate vertical midpoint of the cylinder; the four shocks should be separated from each other by approximately 90° circumferentially.

5.5 Determine \bar{X}_N by counting the particles after the shocks for at least 5 sample intervals, each of at least 6 standard liters (0.2 SCF) or taken over a time of 6 minutes, whichever is greater. The sampling should be completed within 2 hours after the shocks. Calculate X_Q and SE_Q as defined in Section 3.

6 Specification

6.1 Maximum Permissible Particle Concentration — 10 particles per 30 standard liters.

6.2 The specification will be considered met if the calculated concentration of particles plus two standard errors does not exceed 10 particles per 30 standard liters for both measurements before and after the impact shocks, i.e.:

$$X_P + 2SE_P \leq 10 \text{ particles/30 standard liters}$$

and

$$X_Q + 2SE_Q \leq 10 \text{ particles/30 standard liters}$$

7 Report

The report shall contain the value of the measured sample volumes, values of all the variables defined in Section 3, the mass of the steel ball, and length of the chain.

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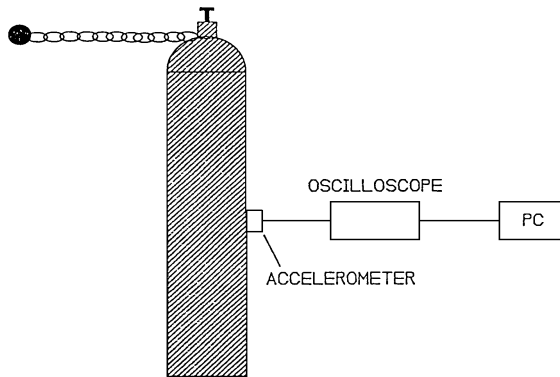


Figure 1

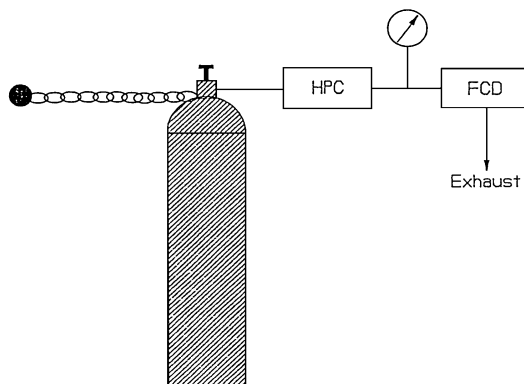


Figure 2

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SEMI C6.2-93 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 20/0.02 OXYGEN

DELIVERED AS PIPELINE GAS

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 1989; previously published in 1993.

1 Purpose

1.1 The purposes of this document are: (1) to set a maximum permissible particle concentration for 20/0.02 grade oxygen bulk supply gas, and (2) to describe a reference method for its verification.

2 Scope

2.1 This document applies only to oxygen gas delivered through pipelines; it is not applicable to cylinder gas or oxygen gas in its liquid state.

2.2 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 Terminology

3.1 Variables

V_{Mi}	Volume of the i^{th} sample interval of the pipeline gas
V_{Bi}	Volume of the i^{th} sample interval of the background
X_{Mi}	Concentration of particles observed in the i^{th} sample interval of the pipeline gas
X_{Bi}	Concentration of particles observed in the i^{th} sample interval of the background
N_M	Number of sample intervals of the pipeline gas
N_B	Number of sample intervals of the background
\bar{X}_M	Average observed concentration of counts in the pipeline gas sample
\bar{X}_B	Average observed concentration of background counts
\bar{X}_C	Calculated concentration of particles in the pipeline gas
S_M	Standard deviation of \bar{X}_M
S_B	Standard deviation of \bar{X}_B
SE_C	Standard error of \bar{X}_C

3.2 *Gas Sample Volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1.00 atmosphere

pressure. Standard Cubic Feet (SCF) is defined at 21.1°C (70°F) and 1.00 atmosphere pressure.

3.3 *Average Observed Concentration of Counts* (\bar{X}_M , \bar{X}_B) — The average concentration of counts, i.e.:

$$\bar{X}_M = \frac{\sum X_{Mi}}{N_M} \quad \bar{X}_B = \frac{\sum X_{Bi}}{N_B}$$

3.4 *Calculated Concentration of Particles* (\bar{X}_C) — The concentration of particles in the pipeline gas obtained by correcting the observed concentration in the pipeline gas for the observed concentration in the background, i.e.:

$$\bar{X}_C = \bar{X}_M - \bar{X}_B$$

3.5 *Standard Deviation* (S_M , S_B , S_C) — A statistical measure of the spread of the concentration of the counts or particles. The first two are obtained from the interval and average concentrations and the number of intervals, i.e.:

$$S_M = \left[\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)} \right]^{1/2} \quad S_B = \left[\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)} \right]^{1/2}$$

The third is obtained from the first two, i.e.:

$$S_C = (S_M^2 + S_B^2)^{1/2}$$

NOTE 1: These expressions are derived from an assumption of a Gaussian (Normal) distribution.

3.6 *Standard Deviation* (S_M , S_B) — A statistical measure of the spread of the concentration of the counts or particles. The first two are obtained from the interval and average concentrations and the number of intervals, i.e.:

$$S_M = \left[\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)} \right]^{1/2} \quad S_B = \left[\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)} \right]^{1/2}$$

NOTE 2: These expressions are derived from an assumption of a Gaussian (Normal) distribution.

3.7 Standard Error (SEC)

$$SE_C = \left[\left(\frac{S_M^2}{N_M} \right) + \left(\frac{S_B^2}{N_M} \right) \right]^{\frac{1}{2}}$$

4 Apparatus

4.1 *Particle Counter* — An instrument suitable for counting particles in gaseous oxygen with a counting efficiency of 50 percent at 0.02 micrometers as determined by the manufacturer of the particle counter. Condensation nucleus counters (CNCs) typically satisfy this requirement.

4.2 *Pressure Reducer* — An accessory required for counters operated at atmospheric pressure, it should preferably use expansion of the gas through a critical orifice.

5 Test Method

NOTE 3: The details of sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice.

5.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing air or nitrogen or oxygen believed to be free of particles of 0.02 micrometers or larger in diameter, through the instrument and recording the total number of counts. Count a minimum of 8 sample intervals, each at least 25 standard liters (0.95 SCF) or 30 minutes, whichever is greater. A suggested assembly for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Calculate \bar{X}_B as defined in Section 3. \bar{X}_B must not exceed 2 particles per 25 standard liters.

5.2 The sampling point should be at outlet of system, and sampling lines should be as short as possible.

5.3 A suggested sampling probe configuration for turbulent main line flow is shown in Figure 2. The flow rate in the sampling tube at pipeline pressure should be set so that the mean sampling flow velocity at the probe inlet matches as closely as possible the axial flow velocity in the pipeline. The pitot sampling tube ID should be no less than 2 mm (0.08 inch). The orifice and sampling horn should be sized so that the mean flow velocity at the particle counter probe inlet matches the axial flow velocity in the horn as closely as possible.

5.4 Count the particles in each of at least 8 sample intervals. Each sample interval must be at least 25 standard liters or 30 minutes, whichever is greater.

Record the number of counts and the sample volume for each interval. Calculate \bar{X}_C and SE_C , as defined in Section 3.

6 Specification

6.1 *Maximum Permissible Particle Concentration* — 20. particles per 25 standard liters as determined by the instrument specified in Section 4.

6.2 The specification will be considered met if the calculated concentration of particles plus two standard errors does not exceed 20. particles per 25 standard liters, i.e.:

$$\bar{X}_C + 2*SE_C \leq 20. \text{ Particles/25 standard liters}$$

7 Report

7.1 The report shall contain the values of all the variables defined in Section 3.

8 Precision

8.1 This test procedure defines the requirements to satisfy the specification at the 95 percent confidence level.

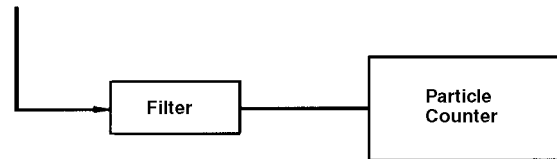


Figure 1
Suggested Assembly for Determining Particle Counter Background

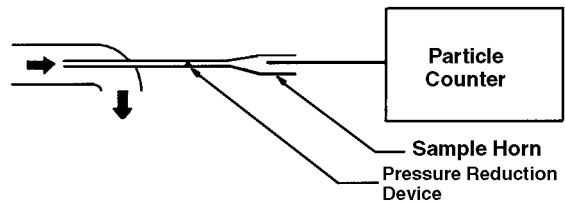


Figure 2
Schematic Diagrams of Configurations of Different Merit for Obtaining Particle Samples from Pipelines



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SEMI C9.1-93 (Reapproved 1102)

GUIDE FOR ANALYSIS OF UNCERTAINTIES IN GRAVIMETRICALLY PREPARED GAS MIXTURES

This guide 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 1993.

1 Purpose

1.1 This document is intended to provide the minimum criterion for the analysis of uncertainty associated with preparation and use of gravimetric gas mixtures used for calibrating analytical instruments to determine whether various SEMI impurity specification are satisfied.

2 Scope

2.1 This guideline is intended for preparation of binary gas mixtures using individual cylinders.

2.2 It is the intent of this document to provide general guidelines for preparation of calibration gas mixtures in compliance with ISO 6142, with additional requirements to meet the needs of the semiconductor industry.

2.3 This guideline is applicable only to gaseous components which do not react between themselves or with the cylinder walls, and to condensable components which are totally vaporized under the test conditions. It is not intended for moisture calibration mixtures.

2.4 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 ISO Standards¹

ISO 6142 — Gas Analysis; Preparation of calibration gas mixtures - weighing methods.

Addendum 1 to ISO 6142 - Annex — Precautions to be taken when weighing, handling and filling cylinders.

4 Weight Traceability

4.1 The weights used to prepare the gas mixtures or certify the balance should be Class S1-tested at least

annually with reference standards traceable to the National Institute of Standards and Technology or to another national standards organization.

5 Acceptable Mixture Criteria

5.1 *Cylinder* — Cylinder must comply with National or International Codes. The type of cylinder used for containment of the gas mixture may affect the stability of the mixture. Historical and experimental data should be requested from the supplier on the stability of similar mixtures. The cylinder material should be chosen based on its compatibility with the gas mixture.

5.2 *Valve* — The recommended valve should be packless type valve and the construction materials selected according to their compatibility with the gas mixture.

5.3 *Homogenous Mixture* — The cylinder and its contents should be at room temperature prior to use. The cylinder mixture components must be mixed to insure a homogenous mixture.

5.4 *Stability* — The gas mixture will have a demonstrated stability within the acceptable relative uncertainties given in Section 6 for a period of 1 year. The minimum useful pressure should be in accordance with the suppliers recommendations.

5.5 *Balance Gas Purity* — The balance gas should be determined to contain less than 1% of the relative concentration for the minor component.

5.6 *Minor Component Purity* — The product used for the minor component addition should be analyzed to verify its composition.

5.7 *Analysis* — Confirmation analysis of the calibration mixture should be performed to determine if any gross weighing errors occurred.

6 Acceptable Relative Uncertainties

6.1 Using the ISO procedures the acceptable maximum relative uncertainties for the component of interest are given below.

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

<i>Concentration Range</i>	<i>Relative Uncertainty</i>
Standard Concentrations > 1000 ppm	2%
Standard Concentrations \leq 1000 ppm > 100	2%
Standard Concentrations \leq 100 ppm > 10	5%
Standard Concentrations \leq 10 ppm \geq 1	10%

7 Report

7.1 The composition of the mixture should be reported including the relative uncertainty for a given mixture.

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SEMI C14-95 (Reapproved 1102)

TEST METHOD FOR PARTICLE SHEDDING PERFORMANCE OF 25 cm GAS FILTER CARTRIDGES

This test method 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 1995; previously published in 1996.

1 Purpose

1.1 The purpose of this document is to define a comprehensive standard test sequence to derive particle-related qualification data for 25 cm (10 inch) filter cartridges.

2 Scope

2.1 This test method defines a particle shedding evaluation method for 25 cm filter cartridges of various media (e.g., PTFE, PVDF, polycarbonate, nylon, and polysulfone) commonly used individually or in assemblies to remove particles from gas lines. The filter cartridges are separable from the housings which can be cleaned and tested independently.

2.2 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 Limitations

3.1 These test methods do not apply to point-of-use (POU) filters, for which separate methods have been developed.

3.2 Cartridge filter performance cannot be characterized by a single data point, as conditions of use (e.g., pressure drop, transient pressure surges) determine the importance of a particular measurement. A sequence of tests, designed to measure filter responses under various conditions, is necessary when evaluating cartridge filters.

3.3 The order of the test sequence will affect the test results, and it is designed for fair comparisons of different aspects of filter operations. Hence, it is important to follow the prescribed test sequence.

4 Summary of Method

4.1 The test sequence (shown schematically in Figure 1) consists of three steps:

1. Break-in
2. Pulse

3. Final Purge

5 Apparatus and Facility

5.1 *Gas Source* — Clean, dry nitrogen or dry air with less than 0.5 ppm moisture and less than 0.5 ppm total hydrocarbons.

5.2 *Particle Detectors* — A condensation nucleus counter (CNC) with a counting efficiency of 50%, as reported by the manufacturer, at 0.01 μm is recommended to measure total particle concentration greater than 0.01 μm .

5.3 *Test Environments* — A clean room environment is not required during testing; however, the test component should be unpacked and assembled in a Class 100 clean area to prevent ambient contamination.

5.4 *Configuration for Break-in Test* — The schematic of the test setup apparatus is shown in Figure 2. All components used in the test apparatus, including the flowmeter, valves, tubing, and filter housing, are 2.5 cm (1 inch) components or have 2.5 cm (1 inch) inlet and outlet connections. The apparatus consists of (1) a differential pressure sensor that can accurately measure Δp in the range 0.01 bar (0.15 psid) to 0.2 bar (3.0 psid) between the upstream and downstream pressure of the test component, (2) a flow control system that permits instantaneous change of flow corresponding to a Δp of 0.01 bar to 0.2 bar across the filter, and (3) a CNC downstream of the test filter.

5.5 *Configuration for Pulse Test* — The schematic of the test setup is the same as the break-in test.

5.6 *Sample Installation* — Reasonable precautions should be taken when installing the test filter or spool piece to avoid particle contamination of the system from ambient air. These precautions may include, but are not limited to, installation in a Class 100 laminar flow area, installation in a purged glove bag or other controlled ambient enclosure, use of a purge flow downstream of the sample point so that all parts of the system are under purge, isolation, or termination of the CNC sample flow, and isolation of the isokinetic sampler exhaust from ambient.

5.7 *Sampling System Design* — Customary practices should be employed for the design of the sampling

system. Care should be taken to provide an adequate exhaust length so that back diffusion of particles does not affect the system background at 2 times the sampling flow rate. This length will depend upon the particle concentration in the ambient environment and other factors.

6 Procedure

6.1 Static Background Test

6.1.1 This test applies to all apparatus described in this document and should be performed prior to other tests.

6.1.2 Remove the filter cartridge from the filter housing and replace the test filter in Figure 2 with the empty housing.

6.1.3 Purge the system at 100 m³/hr (3530 standard cubic feet per hour (scfh)). Measure particle concentration for a minimum of 45 sample intervals, each at least 6 standard liters (0.19 scf) or 4 minutes, whichever is greater. Continue to purge the system until 45 consecutive sample intervals without a particle being detected are recorded.

6.2 Dynamic Background Test

6.2.1 This test applies to the apparatus for pulse test as shown in Figure 2 with the test filter replaced by an empty filter housing.

6.2.2 Purge at a flow rate of 5 m³/hr (177 scfh) until 5 consecutive 60-second sample intervals without a particle being detected are recorded.

6.2.3 Set the CNC sample interval time to 180 seconds. Synchronize the start of this test with the start of a CNC sample interval.

6.2.4 Purge at a flow rate of 5 m³/hr (177 scfh) for 190 seconds.

6.2.5 Instantaneously switch to a flow rate of 100 m³/hr (3530 scfh) by turning ON the solenoid valve and purge for 160 seconds.

6.2.6 Turn OFF the solenoid valve and purge at a flow rate of 5 m³/hr (177 scfh) for 200 seconds.

6.2.7 Repeat Section 6.2.5 and Section 6.2.6 until 20 consecutive pulses without a particle being detected are recorded.

6.3 Break-in Test

6.3.1 The test setup apparatus is shown in Figure 2. Purge at a relatively low flow rate corresponding to a Δp of 0.01 bar (0.15 psid) across the filter until 5 consecutive 60-second sample intervals without a particle being detected are recorded.

6.3.2 Increase the purge flow rate so that the corresponding Δp is at least 0.1 bar and the flow rate is at least 45 m³/hr (1590 scfh).

6.3.3 Record hourly particle concentration averages.

6.3.4 Stop the test when no particles are detected in 3 consecutive hourly sampling intervals.

6.4 Pulse Test

6.4.1 The apparatus is shown in Figure 2. Purge at a relatively low flow rate corresponding to a Δp of 0.01 bar across the filter until 5 consecutive 60-second sample intervals without a particle being detected are recorded.

6.4.2 Set the CNC sample interval to 180 seconds. Synchronize the start of this test with the start of a CNC sample interval.

6.4.3 Purge at a low flow rate corresponding to a Δp of 0.01 bar across the filter for 190 seconds.

6.4.4 Instantaneously switch to a higher flow rate corresponding to a Δp of 0.1 bar (0.15 psid) across the filter by turning ON the solenoid valve and purge for 160 seconds.

6.4.5 Turn OFF the solenoid valve and purge at a low flow rate ($\Delta p = 0.01$ bar) of 200 seconds.

6.4.6 Repeat Section 6.4.4 and Section 6.4.5 (160 secs ON and 200 secs OFF) for a series of 10 flow pulses.

6.5 Final Purge Test

6.5.1 Purge the filter at a Δp of 0.2 bar (3 psid) or a flow rate of 90 m³/hr (3180 scfh), whichever is greater, for 3 hours. Record the particle concentration.

6.5.2 Decrease the flow so that $\Delta p = 0.1$ bar (0.15 psid) or the flow rate is 45 m³/hr (1590 scfh), whichever is greater, and purge for 1 hour. Record the particle concentration.

7 Reporting of Test Results

7.1 Raw data should be reported for each test in the format of a table including the number of sample intervals, the sampling volume of each interval, the sampling time of each interval, and the total number of particles registered in each interval. In addition, the relevant parameters for each test described in Sections 7.2–7.6 should be identified.

7.2 *Static Background Test* — Identify the period of time required to obtain 3 consecutive hourly sampling intervals without a particle being detected.

7.3 *Dynamic Background Test* — Identify period of time required to obtain 20 consecutive flow pulses without a particle being detected.

7.4 *Break-in Test* — Identify the period of time required to obtain 3 consecutive one hour sampling intervals without detection of particles and the total number of particles recorded during this period.

7.5 *Pulse Test* — Identify the number of pulses during which particles were detected and the total number of particle counts.

7.6 *Final Purge Test* — Identify the average particle concentration at each pressure.

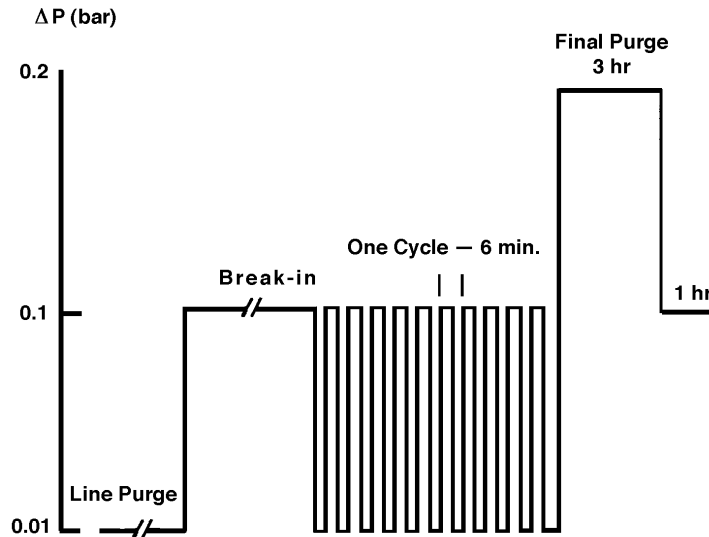


Figure 1
Schematic of a Standard Test Sequence for Evaluating 10-inch Filters

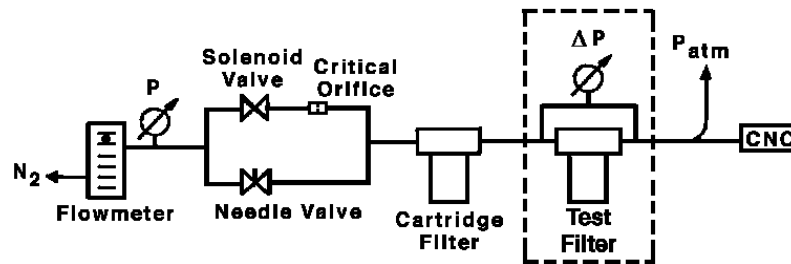


Figure 2
Schematic of the Line Purge Test Setup

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SEMI C15-95 (Reapproved 1102)

TEST METHOD FOR ppm AND ppb HUMIDITY STANDARDS

This test method 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 1995.

1 Purpose

1.1 This test will determine whether or not the quantity of moisture delivered by a gaseous moisture standard is in agreement with that predicted for the standard on the basis of physical principles. It is expected that this method will be used to validate moisture standards before they are first placed in use, when deviation from expected performance is likely or suspected, and at other times as seems necessary.

1.2 The test method assumes the existence of a reliable moisture analyzer. Although extensive precautions are described to ensure the validity of the analyzer, it is always possible that the test could yield a faulty result through some unforeseen defect in the analytical equipment.

1.3 Given that moisture generation is subject to many pitfalls, it will be required that the predicted moisture concentration delivered by the standard and the measurement of that level by the analyzer be independent, and that they agree. The level of agreement can vary depending on the degree of precision claimed for the moisture standard, but in any case the measured and predicted results should be within 10% over the entire range of the moisture standard. If not, then the validation should be repeated after verifying the physical measurement upon which the prediction is based and any other parameters deemed suspect.

2 Scope

2.1 This method is intended to be applicable to any type of gaseous standard delivering a quantity of moisture in the ppm range or lower, provided the delivered moisture concentration is predictable on the basis of fundamental principles of physics. Thus, any standards which rely solely on characterization by analysis are specifically excluded.

2.2 As part of this test, a procedure is described for qualifying certain moisture analyzers considered suitable for use in qualifying moisture standards. Moisture analyzers intended for other applications may need to satisfy different criteria.

2.3 As the most accurate and reliable moisture analyzers currently available operate primarily in the ppm range, this test focuses on validation of moisture

standards in this range. The need for ppb moisture standards is addressed by specifying procedures for reducing the output of ppm moisture standards to the ppb range.

2.4 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 Limitations

3.1 If no higher level of accuracy is specified by the user, then a moisture standard shown to be valid by this method will generate moisture levels which deviate from the expected values by no more than 20%. The method leaves open to the user the option of using sound statistical methods to specify that a given moisture standard will generate levels showing a closer agreement with expected values.

3.2 Validation based on this test method is good for a limited time. The acceptable interval between validations depends on the nature of the standard and is not addressed by this method. Thus any citation of validation results should be accompanied by the date(s) on which validation was performed.

4 Referenced Documents

This method is based upon principles outlined in F. Mermoud, M.D. Brandt, and J.J.F. McAndrew, "Low Level Moisture Generation," *Analytical Chemistry*, 63 (2): 198-202 (1991).

5 Device under Test (Test Specimen)

5.1 *Definition of a Moisture Standard* — A moisture "standard" is defined to be any device capable of delivering a flow of humidified gas at controlled pressure, with no input being provided by the user except nitrogen of UHP grade (moisture < 1 ppm) or air of equivalent purity. Any device, such as a permeation tube, which can provide a predictable amount of moisture but does not include a means to deliver gas to and from it, will not be considered a moisture standard by itself. A device which requires gas of higher purity than UHP grade will include a purifier capable of purifying UHP nitrogen to this level.

5.2 Examples of Moisture Standards — Some examples of moisture standards and the principles upon which they are based are as follows:

- Permeation and effusion tube standards. These must include purification means and some means of regulating the outlet pressure. The moisture delivery rate can be calculated based on the weight loss of the tube.
- Cylinder standards. The “standard” will include a regulator specified for use with the cylinder. The moisture concentration delivered can be calculated based on that added to the cylinder in preparation. A minimum use pressure must be specified.
- Methods based on saturation of gas with water vapor at a fixed temperature and pressure. The moisture concentration in the gas may then be calculated from a knowledge of the saturation vapor pressure of water over a plane of the pure phase of ice at the saturation temperature and of the interaction virial coefficients of the gas-vapor mixture. The two-pressure and two-temperature methods are refinements of this approach requiring additional chambers whose temperature and pressure must be known.

5.3 Dilution of Standards — For all moisture generation methods, lower concentrations can be generated by dynamic dilution (i.e., by combining a known flow of the standard gas with a known flow of dilution gas). A diluted standard is acceptable provided:

- The standard is validated at high concentration (with the lowest concentration validated being no more than 10,000 times the use concentration),
- The dilution gas has a moisture level below the stated precision of the analysis (to be verified using the same flow path as during the subsequent analysis, and at the lowest flow rate to be used),
- The absolute accuracies of the dilution system components are verified by comparison with a reliable flow standard,
- The linearity of the dilution system can be demonstrated over the entire range of operation.

The last criterion is particularly important whenever some portion of the combined flow is discarded, as mixing problems can easily arise at large dilution factors.

6 Procedure

6.1 Validation will be performed using a hygrometer whose output can be directly related to the moisture

concentration on the basis of physical laws or using an analyzer which has recently been calibrated by comparison with such an analyzer.

6.2 Hygrometer Qualification

6.2.1 Primary Hygrometers — Hygrometers can be certified by the National Institute for Standards and Technology (NIST) in the U.S., and similar certification is available in other countries, although the lowest concentration at which certification is available will vary. Repeated certification at yearly or longer intervals can be used to establish reliability. A hygrometer which has been certified by a national standards laboratory to 1 ppm (or lower) moisture in air or nitrogen, and for which reliability has been established over a period greater than or equal to that which has elapsed since certification occurred, is acceptable for validation of a moisture standard and will be referred to as a “primary” hygrometer.

Because hygrometers which are suitable for use as primary hygrometers may be less convenient or less readily available than other hygrometers, the use of other hygrometers for validation is also acceptable, provided adequate precautions are taken to ensure that they are operating correctly. These precautions (to be described below) require using a moisture source to compare a secondary hygrometer with the primary hygrometer. The moisture source need not have been validated in this case.

6.2.2 Validation of Secondary Hygrometers — Secondary hygrometers may be validated by comparing their output with a primary hygrometer when connected in parallel to the same moisture source. The degree of agreement required can vary depending upon the precision to be claimed, but in any case the difference in reading between the two hygrometers should be no more than 10% at any point. Comparison should be made at four moisture concentrations or at two per decade of proposed operation, whichever is less. Half of the comparisons should be made while increasing concentration and half while decreasing, including at least one comparison at the highest point of the concentration range. The first and last comparisons should be at the lowest point of the concentration range. For example, in order to validate a hygrometer for operation between 1 and 100 ppm, it would be acceptable to generate moisture levels at 1, 30, 100, 50, 1 ppm (in that order) and compare the primary with the secondary hygrometer. Comparison of the measured level between the two 1 ppm tests provides a ready indication of hysteresis in the system. If the secondary hygrometer requires calibration using its own standard, calibration should be effected before validation. However, it is acceptable to adjust the calibration in

order to improve agreement with the primary hygrometer.

6.2.3 Frequency of Hygrometer Validation — The frequency of validation required for a secondary hygrometer will vary dependent upon the performance established for hygrometers of the same general type. If three successive monthly validations show continuing agreement (within 10%) with the primary hygrometer without calibration adjustment, the validation interval may be extended to three months. Similar extensions of validation interval to six months and one year may be made if no drift is apparent and no calibration adjustments are necessary to bring the secondary within 10% of the primary at all points.

6.3 Validation Procedure for Moisture Standards

6.3.1 Validation Within the Range of an Available Primary Hygrometer — If a primary hygrometer is available, and its detection limit is no higher than the lowest concentration at which it is desired to use the moisture standard, this is the simplest case. Use the standard to generate four concentration levels or two per decade (whichever is less) in which the standard will be used, half of the concentrations generated while increasing concentration, half while decreasing, including at least one comparison at the highest point of the concentration range. The first and last comparisons should be at the lowest point of the concentration range. If the generated levels, calculated according to the basic properties of the generator (e.g., weight loss of a permeation tube) agree with the measured levels to within 10%, the standard may be considered validated with an accuracy of 10%. Higher validation accuracy requires better agreement. It has been assumed that the precision of the primary hygrometer, as determined by a national standards laboratory, is much better than 10% and may be neglected for validation at the 10% level. Validation at higher accuracy requires explicit consideration of the certification precision.

If a standard only generates one level, connect it to the hygrometer and allow to come to equilibrium. Assuming the equilibrium level agrees with the calculated level of the generator, disconnect the hygrometer and bring it to equilibrium with either dry nitrogen or ambient air. Then reconnect the moisture standard and reestablish equilibrium between the standard and the hygrometer. Disconnect again and repeat a third time. If the three measurements agree within 10% among themselves and with the generated level, the standard is validated.

It is implicit in the above that the background moisture level of the system be less than 50 ppb so that it can be neglected for all measurements above 1 ppm with an error no larger than 5%. We can usually neglect

background moisture in validating moisture standards in this concentration range.

6.3.2 Validation Using Secondary Hygrometers — If no primary hygrometer is available, then validation can be performed using a secondary hygrometer. If the lowest concentration to be generated using the standard is no higher than the detection limit of the primary hygrometer used for validation of the secondary hygrometer, then the procedure is as above, except that the secondary must first have been validated over the entire concentration range which the standard will be used to generate. (A secondary hygrometer and a moisture standard can be validated simultaneously if the primary and secondary hygrometers are connected in parallel to the moisture standard.)

When assessing the accuracy of validation using a secondary hygrometer, it is necessary to take into account the precision of the comparison of the primary hygrometer with the secondary hygrometer as well as the precision of the comparison of the generator with the secondary hygrometer.

7 Practice for Generating ppb Moisture Standards

The detection limit of the primary hygrometer will generally be on the order of 1 ppm, whereas it will frequently be necessary to use the moisture standard at concentrations below this level, by a factor of as much as 10,000. Therefore, valid methods for reducing concentration are necessary.

7.1 Dilution of Validated Moisture Standards — If a moisture standard validated in accordance with Section 6.2 or Section 6.2.1 is diluted with a zero gas stream satisfying the criteria of Section 5.3, then the resultant diluted standard may be considered valid and may be used for calibration of analyzers, such as APIMS.

7.2 Consideration of Background Moisture — In any ppb or lower moisture measurement, it is necessary to consider the background moisture level due to outgassing in the sampling system and residual moisture in the generator + analyzer. Often the background will be sufficiently large that it must be taken into account explicitly. The background moisture level is the moisture concentration measured in the absence of a moisture source (i.e., when the driest carrier gas available is fed to the analyzer using the same flow rate and flow path as is used for the sample gas during analysis).

It should be noted that the importance of the background level increases for measurements at low concentrations, and that the background level is sensitive to the materials of construction, to the design

of the moisture generator, and to its operating temperature.

If the background moisture concentration is B (ppb), the concentration delivered by the moisture standard is C (ppb) and the dilution factor x is given by

$$x = \frac{\text{flow of moisture standard}}{\text{total flow}} \quad (1)$$

then c , the delivered moisture concentration is given (in ppb) by

$$c = (1 - x)B + xC \quad (2)$$

The accuracy of calibration will reflect the combined accuracy of the standard and the dilution system, together with the accuracy of the background determination; all must be taken into account in any statement of analytical uncertainty.

7.3 Changing the Range of Performance of a Moisture Standard — If a moisture standard is based on some device which is believed to deliver moisture at a constant rate, such as a permeation or effusion tube, the concentration range delivered by the moisture standard may be changed by delivering a substantially different flow of carrier gas. This usually requires changing a mass flow controller or other flow control device inside the standard for a flow controller of a different range. This may be done provided the new flow controller's performance is verified by comparison with a reliable flow standard.

The definition of dilution factor is revised to

$$x = \frac{\text{flow generated by original flow controller}}{\text{flow generated by new flow controller}} \quad (3)$$

The background, B , is determined by removing the permeation or effusion tube from the system and capping off its point of connection, or by preventing the source from delivering moisture by some other means. The same equation for c applies as does the statement regarding analytical uncertainty.

7.4 Validation at Intermediate Concentrations — It may be desirable to validate a moisture standard at a concentration below the detection limit of the primary hygrometer, if that standard is to be used for very low-level generation. For example, if a standard based on a permeation device is intended to generate levels between 0.5 and 5 ppb at flows of 1 to 10 slm, it could be validated in the 0.5 to 5 ppm range using a frost-point hygrometer by replacing the flow controller with

one operating between 1 and 10 sccm¹. However, such a flow controller may not be readily available and is difficult to calibrate in any case. Instead, it is preferable to use a flow controller operating between 50 and 500 sccm to generate concentrations between 0.1 and 1.0 ppm together with a secondary hygrometer having a detection limit no higher than 50 ppb. This procedure is acceptable, provided that

- the secondary hygrometer has been validated with a primary hygrometer at least at one point in the range to be used for validation of the moisture standard,
- the moisture concentrations generated by the standard and that observed by the secondary hygrometer agree throughout the range of validation of the standard.

This approach could lead to errors if the hygrometer and the standard each exhibited a deviation from predicted performance, but these deviations would have to be the same and cancel, which is extremely unlikely.

In the case of operation in this mode, it will be necessary to know the background level of the system and use this as an input to the calculation of generated moisture levels. To continue the example of a permeation based system, if the permeation rate determined by weight loss is P (ng/min), the flow rate of carrier gas is F (sccm), K is a factor to convert ng/min to sccm moisture, and B is the experimentally determined background, then the generated moisture level, c_{gen} , is calculated according to

$$c_{gen} = \frac{KP}{F} + B \quad (4)$$

For other moisture generation systems, it is similarly possible to calculate the generated moisture level while taking into account the background level of the system. This assumes that the background moisture concentration is constant, which is only approximately true. Thus, in applying this approach, in addition to the sources of error mentioned above, the estimate of analytical uncertainty must also include the variation in background moisture.

8 Validation Precision and Accuracy

This procedure does not explicitly consider the estimation of the accuracy of a moisture standard in

¹ It is assumed that, although flow controllers are calibrated using a standard, no unusual measures are used to enable the full accuracy of calibration to be achieved over the entire range of the flow controller. In practice this means that a flow controller has a usable dynamic range of 10-100% of its flow rating.

detail, because such a calculation can be made by applying procedures described elsewhere. The accuracy required in validation will vary with the proposed application of the moisture standard and may be left to the discretion of the user of the procedure. It is recommended that a propagation of errors calculation be carried out to estimate the accuracy of the validation. If no such calculation is made, and the minimum criteria of the procedure are followed, it is estimated that a validated moisture generator may output moisture levels which differ from the true values by up to 20%.

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SEMI C52-0301

SPECIFICATION FOR THE SHELF LIFE OF A SPECIALTY GAS

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the European Gases Committee. Current edition approved by the European Regional Standards Committee on December 20, 2000. Initially available at www.semi.org January 2001; to be published March 2001.

1 Purpose

1.1 The purpose of this document is to define terminology and to recommend minimum periods for the shelf lives of specialty gases. It is meant to provide consistency in terminology among gas suppliers and to provide a general guideline for users of these gases.

2 Scope

2.1 This document applies to the shelf life of properly packaged, filled and analyzed specialty gases as stored or supplied by a specialty gas manufacturer or supplier. In this document, shelf life is viewed from a quality point of view. The document does not address safety aspects associated with the prolonged storage of gases.

2.2 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 limitations prior to use.

3 Referenced Standards

None.

4 Terminology

4.1 *container* — a lecture bottle, cylinder, cylinder pack, drum or any other vessel which is used to supply a specialty gas.

4.2 *residual level* — The amount of product, as a fraction of the fill weight or fill pressure, which should be left in a container in order to minimize the impact on manufacturing processes of the higher level of impurities in the last gas fraction from a container.

4.3 *shelf life* — the period of time for which the specification of a gas is guaranteed by the supplier, starting from the time of analysis. It defines the period for which the supplier guarantees the actual level of impurities, as analyzed, to remain at or below the specification limit for a particular gas grade.

5 Requirements

5.1 *Factors Affecting the Shelf Life of a Specialty Gas* — Shelf life is a function of the following factors:

a. Thermal stability of the gas

b. Quality of the container and valve

- material selection
- leak tightness

c. Time-dependent reactions between

- gas and impurities
- gas and packaging materials

d. Filling and analytical operations

e. Transport and storage conditions

f. Amount of product in the container

5.1.1 The integrity of the valve is a key factor determining the shelf life of a gas, in particular for low vapor pressure gases. Time dependent reactions can be significantly reduced by purification of the gas to a level where impurities no longer have a significant effect over time, and by selection of proper packaging materials. Chemical and physical treatment of the internal container surface and/or passivation of this surface before filling also reduces the effect of time-dependent reactions. For material and gas compatibility information, please refer to the individual SEMI gas standards.

5.2 *Classification of Specialty Gases on the Basis of Shelf Life* — While there are a large number of specialty gases used in semiconductor manufacturing, for the purpose of defining shelf lives, they can be divided into four groups. It should be noted that for example chemically reactive gases are sometimes classified as non-reactive in the table below as the classification is done from a shelf-life point of view only.

5.2.1 *Non-reactive* — Under normal storage conditions, these gases do not react with ambient air, the packaging materials or the impurities commonly found in the specialty gas.

5.2.2 *Reactive* — These gases react either with ambient air or standard packaging materials.

5.2.3 *Corrosive* — These gases react with packaging materials usually in the presence of traces of moisture.

5.2.4 *Unstable* — These gases are thermally unstable and spontaneously decompose over time.

Table 1 Classification of Specialty Gases on the Basis of Shelf Life

<i>Non-reactive</i>	<i>Reactive</i>	<i>Corrosive</i>	<i>Unstable</i>
Air	AsH ₃	BCl ₃	B ₂ H ₆
Ar	CO	BF ₃	NO
CFH ₃	D ₂	¹¹ BF ₃	
CF ₂ H ₂	GeH ₄	ClF ₃	
CF ₃ H	H ₂	Cl ₂	
CF ₄	NF ₃	F ₂	
CH ₄	PH ₃	GeF ₄	
CO ₂	SeH ₂	HBr	
C ₂ F ₆	SiH ₄	HCl	
C ₃ F ₈	Si ₂ H ₆	HF	
C ₄ F ₈		NH ₃	
C ₅ F ₈		PF ₅	
He		SiCl ₂ H ₂	
Kr		SiCl ₃ H	
Ne		SiCl ₄	
N ₂		SiF ₄	
N ₂ O		WF ₆	
O ₂			
SF ₆			
Xe			

5.3 Guaranteed Shelf Life Period — The following guaranteed shelf life periods are recommended as a minimum.

Table 2 Guaranteed Shelf Life Period

<i>Non-reactive</i>	<i>Reactive</i>	<i>Corrosive</i>	<i>Unstable</i>
36 months	24 months	18 months	6 months

5.3.1 When proper packaging materials are chosen and the container is properly prepared (and passivated), impurity levels should in theory remain constant over time for an electronics grade specialty gas with the exception of the thermally unstable gases. For this reason, no maximum shelf life periods are recommended as these will depend on the level of analytical data collected by the manufacturer or supplier.

5.4 Mixtures — The shelf life of a mixture is recommended to be equal to the shelf life of the component in the mixture with the lowest shelf life. For example, the minimum shelf life of a reactive gas in a non-reactive gas matrix would be 24 months. The shelf life of a mixture should apply both to the impurity specifications and to the assay of the mixture.

5.4.1 Special attention should be paid to the preparation of the package for mixtures containing less than 1,000 ppm of a component. At such levels, adsorption effects may reduce the shelf life and minimization of the package internal surface roughness and/or passivation of the surface may be required.

5.5 Residual Level — The impact of the depletion of a container on impurity levels is still a matter of debate among gas suppliers. In general, the impurity levels of the first gas fraction from a container will differ from the last gas fraction. In particular moisture levels tend to rise at lower gas pressures as a result of the relative increase in importance of the internal container wall and its adsorbed moisture. In addition, in liquefied gases the co-existence of both a gas phase and a liquid phase can have a measurable effect on the distribution of impurities over the gas fractions. It is argued that at the point of liquid-dry some impurities, which prefer solution in the liquid phase over the gas phase, may see a dramatic rise. It is therefore recommended to always leave an amount of product in the container and to avoid a situation of “liquid dry” (i.e., when all of the liquid phase of a liquefied gas has been used).

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SEMI C54-1103

SPECIFICATIONS AND GUIDELINES FOR OXYGEN

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 27, 2003. Initially available at www.semi.org October 2003; to be published November 2003.

1 Purpose

1.1 The purpose of this document is to provide a series of specifications for different grades of Oxygen (O₂) 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: 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 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 O₂ 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 — Specifications for Reagents

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

<i>Previous SEMI Reference #</i>	<i>C3.22-1000 (Specification)</i>	<i>C3.23-1000 (Specification)</i>	<i>C3.41-0697 (Specification)</i>
Grade	2.5	3.8	5.8
Purity	99.5%	99.98%	99.9998% (See Note 1.)
Impurities	Maximum Acceptable Level (ppm) (See Note 2.)		
Carbon Dioxide and Carbon Monoxide (CO ₂ + CO)	5	N/A	N/A
Carbon Dioxide (CO ₂)	N/A	1	0.1
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 ₂ O)	2	1	N/A
Argon (Ar)	N/A	100	1.0
Krypton (Kr)	N/A	10	N/A
Water (H ₂ O) (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	(See Note 3)	(See Note 3)	(See Note 3)

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

Note 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.

Note 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)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	31.999	31.999
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

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 Section 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 Section 7 of this standard.

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

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 Section 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 Section 7 of this standard.

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

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, 4.)

NOTE 2: The 0-1 range can be used provided that zero and span gas standards in oxygen with known levels of hydrocarbons between 0-1 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.

9.3.2 *Flow Requirements*

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

9.3.2.2 Dry, hydrocarbon-free (less than 1.0 ppm) air: 350–400 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 CH₄ level must not exceed 4 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 0–10 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 Section 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, 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, e.g., 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 prepurging 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.

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 Section 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*:

- 1-100 mL calibrated certified burette, calibrated every 0.1 mL for the top 5 mL equipped with a 3-way stopcock on the top.
- 3-250 mL aspirator bottles.
- 1 one-hole rubber stop.

- 1-1/16 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 #1, through the copper coil bottle #3, and the gas burette while, at the same time, 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 #1 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 Notes 1 in 9.2.4.1 and 9)

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: 1–3 mL.

10.1.2.4 Temperatures:

Detector	40°C
Column	–50°C

10.1.3 *Calibration Standard* — 50–150 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 Section 10.1.4.1. Record the retention times and peak areas.

10.1.4.3 Repeat Section 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 Section 7 of this standard.

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

10.2 *Carbon Monoxide* — This procedure is for the determination of carbon monoxide using dual beam optical non-dispersive infrared spectrophotometry.

10.2.1 *Detection Limit* — 0.2 ppm.

10.2.2 *Instrument Parameters*

10.2.2.1 Detector: “Luft” type or equivalent

10.2.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

10.2.2.3 Sample Cell Pressure: 200 psig for full scale range 0–20 ppm carbon monoxide or appropriate pressure recommended by the cell manufacturer.

10.2.2.4 Sample Flowrate: 500 cc/minute or as specified by the instrument manufacturer.

10.2.2.5 Wavelength: 4.75 micrometers (4750 nm)

10.2.2.6 Wavenumber: 2100 cm^{-1}

10.2.3 *Calibration Standard* — 5 ppm carbon monoxide, balance oxygen.

10.2.4 *Operating Procedure* (See Notes 10, 11) (Refer to Figure 4).

NOTE 10: An example of an operating procedure is outlined below for the sample system shown in Figure 3. This procedure is appropriate only when the infrared gas cell is designed to withstand the 200 psig sample pressure.

NOTE 11: Operating procedures for other non-dispersive infrared analyzers vary depending on manufacturer. Refer to individual instrument vendor instructions in each case.

10.2.4.1 Open the zero gas (pre-purified nitrogen or certified pure oxygen, independently measured to be less than 0.1 ppm CO) cylinder valve. Open valve V1, close valves V2 and V3. Flow the gas through the system and adjust the back pressure regulator to 200 psig as shown on gauge G. Adjust the flowrate on the flowmeter to 1000 cc/minute by adjusting valve V1. After a constant readout is observed, adjust the zero control knob of the analyzer to set the absorbance output to read zero.

10.2.4.2 Open the calibration gas standard cylinder valve. Close V1 and V2, and open valve V3. Flow the calibration gas through the system and adjust the back pressure regulator to 200 psig. Adjust the flowrate on the flowmeter to 1000 cc/minute. After a constant readout is observed, record the absorbance of the calibration standard, if the instrument indicates absorbance directly. If the instrument indicates concentration, adjust the span control to read the concentration of carbon monoxide in the calibration gas.

10.2.4.3 Introduce the oxygen sample into the analyzer by closing valves V1 and V3, opening valve V4 and slowly opening valve V2 until the flowrate on the flowmeter is 1000 cc/minute. Adjust the back pressure regulator to 200 psig. If the instrument indicates absorbance, read the absorbance of the sample and calculate the quantity of carbon monoxide, using the formula below. If the instrument indicates concentration, record the concentration. The results may not exceed the specification in Section 7 of this standard.

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

10.3 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide using dual beam optical non-dispersive infrared spectrophotometry.

10.3.1 *Detection Limit* — 0.05 ppm.

10.3.2 *Instrument Parameters*

10.3.2.1 Detector: “Luft” type or equivalent

10.3.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

10.3.2.3 *Sample Cell Pressure* — 100 psig for full scale range 0–5 ppm carbon dioxide; 200 psig for full scale range of 0–2.5 ppm carbon dioxide or appropriate pressure as recommended by cell manufacturer.

10.3.2.4 Sample Flowrate: 500 cc/minute or as specified by the instrument manufacturer

10.3.2.5 Wavelength: 4.4 micrometers (4400 nm)

10.3.2.6 Wavenumber: 2250 cm^{-1}

10.3.3 *Calibration Standard* — 5 ppm carbon dioxide, balance oxygen.

10.3.4 *Operating Procedure* — (See Notes 10, 11 in 10.2.4.) (Refer to Figure 4.)

10.3.4.1 Open the zero gas (prepurified nitrogen or pure oxygen, independently measured to be less than 0.1 ppm CO₂) cylinder valve. Open valve V1, close valves V2 and V3. Flow the gas through the system and adjust the back pressure regulator to 100 psig as shown on gauge G. Adjust the flowrate on the flowmeter to 1000 cc/minute by adjusting valve V1. After a constant readout is observed, adjust the zero control knob of the analyzer to set the absorbance output to read zero.

10.3.4.2 Open the calibration gas standard cylinder valve. Close V1 and V2, and open valve V3. Flow the calibration gas through the system and adjust the back pressure regulator to 100 psig. Adjust the flowrate on the flowmeter to 1000 cc/minute. After a constant readout is observed, record the absorbance of the calibration standard, if the instrument indicates absorbance directly. If the instrument indicates concentration, adjust the span control to read the concentration of carbon dioxide in the calibration gas.

10.3.4.3 Introduce the oxygen sample into the analyzer by closing valves V1 and V3, opening valve V4 and slowly opening valve V2 until the flowrate on the flowmeter is 1000 cc/minute. Adjust the back pressure regulator to 100 psig. If the instrument indicates absorbance, read the absorbance of the sample and calculate the quantity of carbon dioxide, using the formula below. If the instrument indicates concentration, record the concentration. The results

may not exceed the specification in Section 7 of this standard.

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

10.4 Krypton and Nitrogen — This procedure is for the determination of krypton and nitrogen using a gas chromatograph with a thermal conductivity detector.

10.4.1 Detection Limits — 3 ppm krypton, 3 ppm nitrogen.

10.4.2 Instrument Parameter

10.4.2.1 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.

10.4.2.2 Carrier Flow: 45 mL/min helium.

10.4.2.3 Sample Volume: 25 mL.

10.4.2.4 Temperatures:

Detector	40°C
Column	25°C

10.4.3 Calibration Standards — 5–15 ppm krypton, 5–15 ppm nitrogen, balance helium.

10.4.4 Operating Procedure (Refer to Figure 5)

10.4.4.1 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 and allow the oxygen to pass through the column to vent. Switch valve to Position A to allow the krypton and nitrogen to be carried to the detector. Record the peak areas and retention times.

10.4.4.2 Inject the sample to be tested in the same manner as Section 10.4.4.1. Record the retention times and peak areas.

10.4.4.3 Repeat 10.4.4.2.

10.4.4.4 Calculate the concentrations of krypton and nitrogen using the formula below. The result may not exceed the specification in Section 7 of this standard.

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

10.5 Nitrous Oxide — This procedure is for the determination of nitrous oxide using dual beam optical non-dispersive infrared spectrophotometry.

10.5.1 Detection Limit — 0.05 ppm.

10.5.2 Instrument Parameters

10.5.2.1 Detector: “Luft” type or equivalent

10.5.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

10.5.2.3 Sample Cell Pressure: 200 psig for full scale range 0–5 ppm nitrous oxide or appropriate pressure as recommended by the cell manufacturer.

10.5.2.4 Sample Flowrate: 500 cc/minute or as recommended by the instrument manufacturer

10.5.2.5 Wavelength: 4.5 micrometers (4500 nm)

10.5.2.6 Wavenumber: 2222 cm⁻¹

10.5.3 Calibration Standard: 5 ppm nitrous oxide, balance oxygen.

10.5.4 Operating Procedure — (See Notes 10, 11 in Section 10.2.4.) (Refer to Figure 4.)

10.5.4.1 Open the zero gas (prepurified nitrogen or certified pure oxygen, independently measured to be less than 0.1 ppm N₂O) cylinder valve. Open valve V1, close valves V2 and V3. Flow the gas through the system and adjust the back pressure regulator to 200 psig as shown on gauge G. Adjust the flowrate on the flowmeter to 1000 cc/minute by adjusting valve V1. After a constant readout is observed, adjust the zero control knob of the analyzer to set the absorbance output to read zero.

10.5.4.2 Open the calibration gas standard cylinder valve. Close V1 and V2, and open valve V3. Flow the calibration gas through the system and adjust the back pressure regulator to 200 psig. Adjust the flowrate on the flowmeter to 1000 cc/minute. After a constant readout is observed, record the absorbance of the calibration standard, if the instrument indicates absorbance directly. If the instrument indicates concentration, adjust the span control to read the concentration of nitrous oxide in the calibration gas.

10.5.4.3 Introduce the oxygen sample into the analyzer by closing valves V1 and V3, opening Valve V4 and slowly opening valve V2 until the flowrate on the flowmeter is 1000 cc/minute. Adjust the back pressure regulator to 200 psig. Read the absorbance of the sample and calculate the quantity of nitrous oxide, using the formula below. If the instrument indicates concentration, record the concentration. The result may not exceed the specification in Section 7 of this standard.

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

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

10.6.1 Detection Limit — 0.1 ppm.

10.6.2 Flow Requirements

10.6.2.1 High purity, hydrocarbon-free (less than 0.1 ppm) hydrogen, 35–40 mL/min or 40% hydrogen in either helium or nitrogen at 75–80 mL/min.

10.6.2.2 Dry, hydrocarbon-free (less than 0.1 ppm) air, 350 mL–400 mL/min.

10.6.2.3 Set sample flow rate in accordance with the instrument manufacturer's instructions.

10.6.3 Calibration Standards

10.6.3.1 Zero oxygen with a known quantity of hydrocarbons of approximately 0.1 ppm.

10.6.3.2 Upper level balance oxygen span gas of CH₄ that is not more than five times the concentration specified in Section 7 of this Standard.

10.6.4 Operating Procedure

10.6.4.1 Do not change initial flow settings for hydrogen, air and sample once established.

10.6.4.2 Introduce the zero oxygen with a known quantity of hydrocarbons and, using the 0–1 ppm range, set the output to read the correct level, using the zero adjust knob.

10.6.4.3 Introduce the span gas standard in oxygen and, using the span adjust knob, set the output to the level in the span gas.

10.6.4.4 Introduce the oxygen sample into the analyzer and read the quantity of hydrocarbons on the analyzer. The result may not exceed the specification in Section 2 of this standard.

10.7 Water — This procedure is for the determination of trace moisture (water) in oxygen using a continuous flowing, cooled-surface condensation, dewpoint/frostpoint hygrometer. (See Notes 5, 6, 7 in Section 9.4.)

10.7.1 Detection Limit — 0.6 ppm at –79°C(–110°F)

10.7.2 Flow Requirements

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

10.7.3 Calibration Standard — A calibration thermometer designed to indicate temperatures in the –79°C(–110°F) range is required.

10.7.4 Operating Procedure

10.7.4.1 An appropriately cleaned stainless steel or copper sampling line must be used. If it has been exposed to ambient moisture, it must be purged with dry gas prior to use.

10.7.4.2 Flow the sample gas through the hygrometer until the sampling system and instrument have reached equilibrium with the gas.

10.7.4.3 After equilibrium has been reached, cool the mirror of the hygrometer, as specified by its manufacturer, to determine the dewpoint/frostpoint of the sample gas.

10.7.4.4 Verify the dewpoint/frostpoint reading for at least 30 minutes after it becomes stable.

10.7.4.5 Correct the dewpoint/frostpoint reading to 1 atm pressure. Convert it to ppm (vol/vol) using an appropriate table. The result may not exceed the specification in Section 7 of this standard.

11 Analytical Procedures for Grade 5.8 Oxygen

11.1 Argon and Nitrogen — This procedure is for the determination of argon and nitrogen in oxygen using a gas chromatograph with a discharge ionization detector (see Figure 6).

11.1.1 Detection Limit — 0.1 ppm

11.1.2 Instrument Parameters

11.1.2.1 Column(s):

3 m (10 ft) by 3.2 mm (1/8") o.d. in Molecular Sieve 5A or equivalent.

3 m (10 ft) by 1/8 in Oxy-trap (See Figure 6)

11.1.2.2 Carrier Flow: 44 cc/min

11.1.3 Sample Volume: 0.1 mL

11.1.3.1 Temperatures:

Detector	100°C
Column	30°C
Oxytrap	150°C

11.1.4 Calibration Standards — 1–5 ppm nitrogen and argon in oxygen.

11.1.5 Operating Procedures

11.1.5.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak area. Order of elution is argon, nitrogen. If methane is present, the order is argon, nitrogen, methane.

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

11.1.5.3 Repeat Section 11.1.5.2.

11.1.5.4 Compare the average peak areas of the calibration standard to that of the sample being tested. Calculate the concentrations of impurities as shown in SEMI C3. The result may not exceed the specification in Section 7 of this standard (see Note 12).

NOTE 12: Oxygen trap conditioned per manufacturer's recommendations using hydrogen, carbon monoxide, or blend in helium.

11.2 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in oxygen using a gas chromatograph with a flame ionization detector and a methanizer.

11.2.1 *Detection Limit* — 0.02 ppm

11.2.2 *Instrument Parameters*

11.2.3 Column(s): 2.3 m (91 in) by 1/8 in HayeSep Db

11.2.3.1 Carrier Flow: 50 cc/min nitrogen

11.2.3.2 Sample Volume: 5 mL

11.2.3.3 Temperatures:

Detector	275°C
Column	50°C
Methanizer	295°C

11.2.4 *Calibration Standards* — 0.1–1 ppm carbon dioxide in oxygen.

11.2.5 *Operating Procedures* (See Note 13.)

NOTE 13: Sample and calibration sample are injected on column to vent until oxygen, carbon monoxide, and methane have eluted. Valve is returned to allow carbon dioxide to elute onto methanizer and flame ionization detector.

11.2.5.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak area. Order of elution is: Oxygen + carbon monoxide, methane, carbon dioxide.

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

11.2.5.3 Repeat Section 11.1.10.2.

11.2.5.4 Compare the average peak areas of the calibration standard to that of the sample being tested. Calculate the concentrations of impurities as shown in SEMI C3. The result may not exceed the specification in Section 7 of this standard (see Note 17).

11.3 *Carbon Monoxide and Hydrogen* — This procedure is for the determination of carbon monoxide and hydrogen in oxygen using a gas chromatograph with a reduction gas detector.

11.3.1 *Detection Limit* — 0.002 ppm

11.3.2 *Instrument Parameters*

11.3.2.1 Column(s): 2 m (80 in) by 1/8 in Molecular Sieve 13×

11.3.2.2 Carrier Flow: 20 cc/min air

11.3.2.3 Sample Volume: 1 mL

11.3.2.4 Temperatures:

Detector	265°C
Column	105°C

11.3.3 *Calibration Standards* — 0.5 – 1 ppm hydrogen and carbon monoxide in nitrogen or oxygen.

11.3.4 *Operating Procedures*

11.3.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak area. Order of elution is: Hydrogen, carbon monoxide.

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

11.3.4.3 Repeat Section 11.2.4.2.

11.3.4.4 Compare the average peak areas of the calibration standard to that of the sample being tested. Calculate the concentrations of impurities as shown in SEMI C3. The result may not exceed the specification in Section 7 of this standard.

11.4 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in oxygen with a total hydrocarbon analyzer.

11.4.1 *Detection Limit* — 0.05 ppm

11.4.2 *Instrument Parameters*

11.4.2.1 Flow Requirements:

11.4.2.1.1 Carrier Flow Rate: per manufacturer's recommendations

11.4.2.1.2 Purity: less than 0.05 ppm total hydrocarbons

11.4.2.1.3 Sample Flow Rate: manufacturer's recommendations

11.4.3 *Calibration Standards* — 0.1–1 ppm methane in oxygen.

11.4.4 *Operating Procedures*

11.4.4.1 Flow settings should not be changed once established.

11.4.4.2 Introduce the zero gas and adjust the instrument per manufacturer's recommendations to read the quantity known to be in the zero gas.

11.4.4.3 Introduce the span gas standard and adjust the instrument per manufacturer's recommendations to read the quantity known to be in the span gas. This result may not exceed specification in Section 7 of this standard.

11.5 *Water* — This procedure is for the determination of trace moisture (water) in O₂ using a continuous flowing piezoelectric hygrometer (see Note 5, 6, 7 in Section 9.4).

11.5.1 *Detection Limit* — 5 ppb

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

11.5.3 *Calibration Standards* — Construct a calibration curve that contains at least three points covering the range of interest. Verify the standards employed independently by another analytical method.

11.5.4 *Operating Procedure*

11.5.4.1 Obtain a continuous flow sample of gas from the source using a clean and passivated stainless steel line that has been purged dry after exposure to ambient moisture.

11.5.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and the piezoelectric moisture hygrometer until a stable reading is obtained.

11.5.4.3 Determine the moisture content of the oxygen sample by comparing the reading to calibration curve. The result may not exceed the specification in Section 7 of this standard.

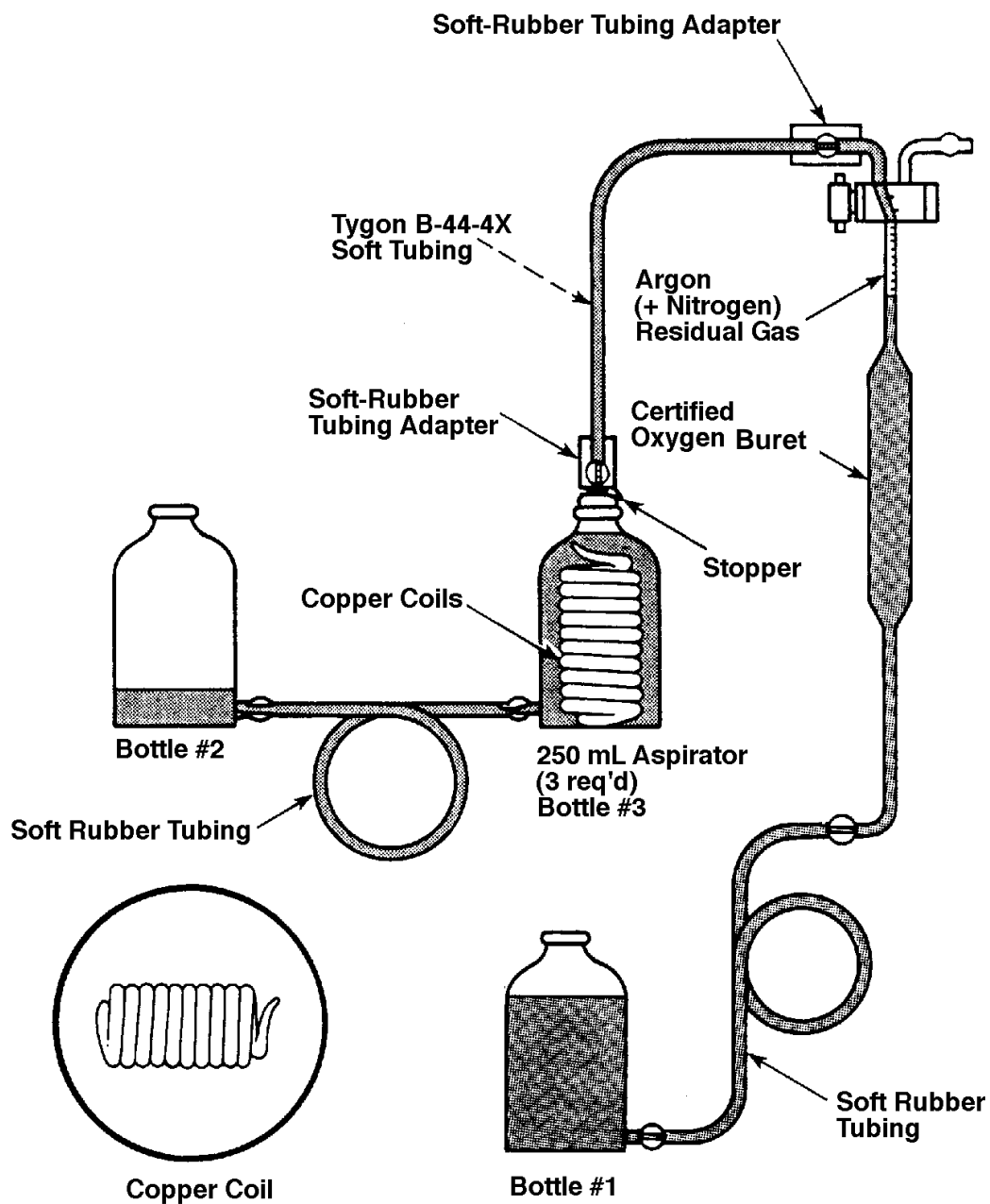


Figure 1
"Orsat" Device for the Assay Of O_2

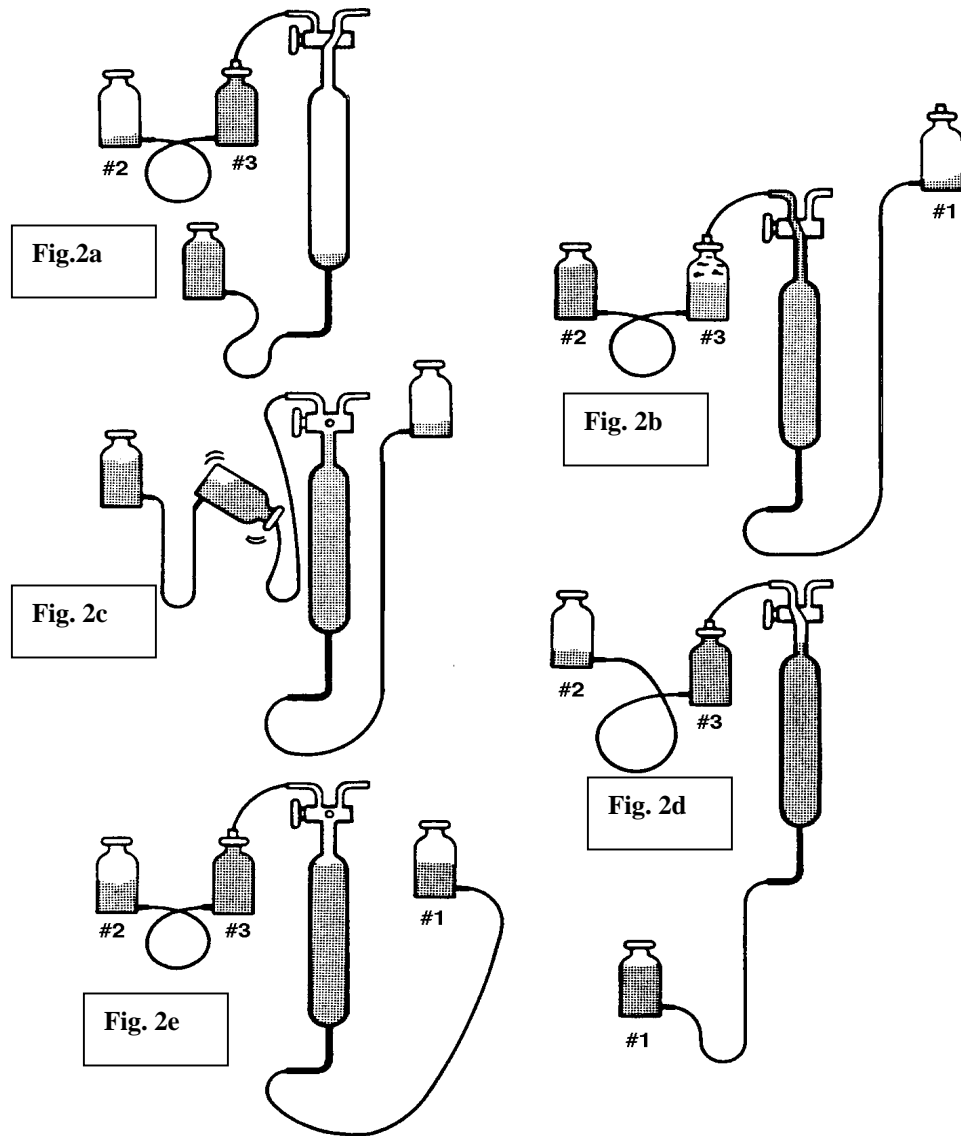


Figure 2
Relative Position of the Different Vessels for the “Orsat” Device for the Assay of O₂

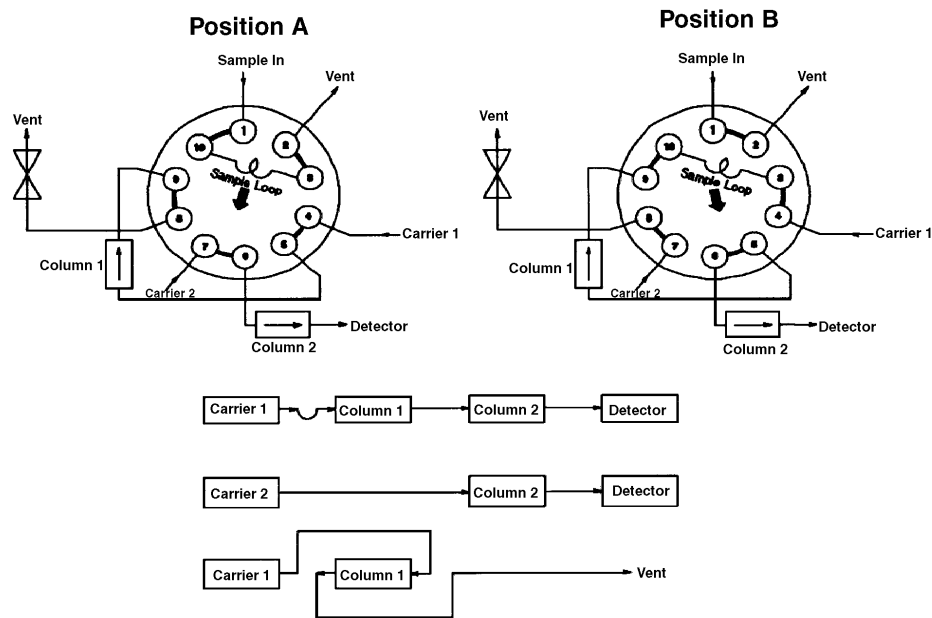


Figure 3
Valve Sampling System for the Analysis of Ar in Grade 3.8

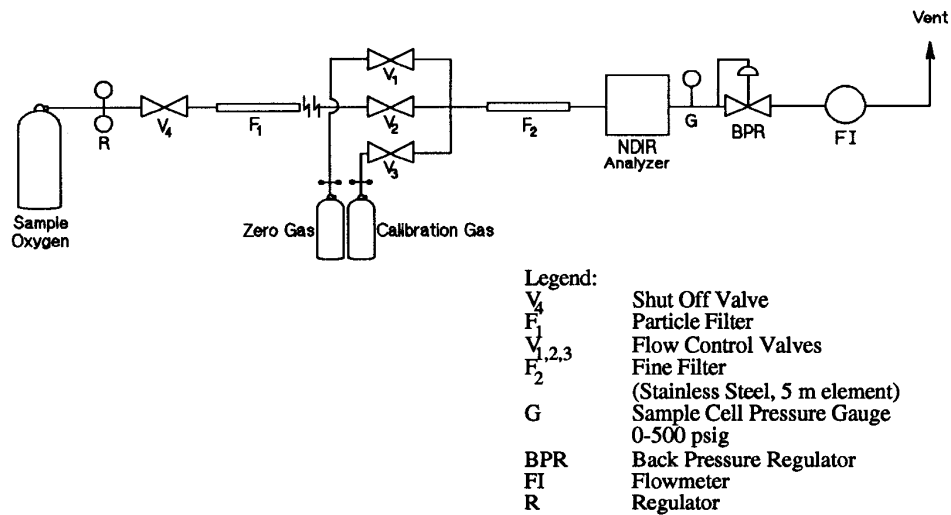


Figure 4
Sample System for NDIR Analyzer in Grade 3.8

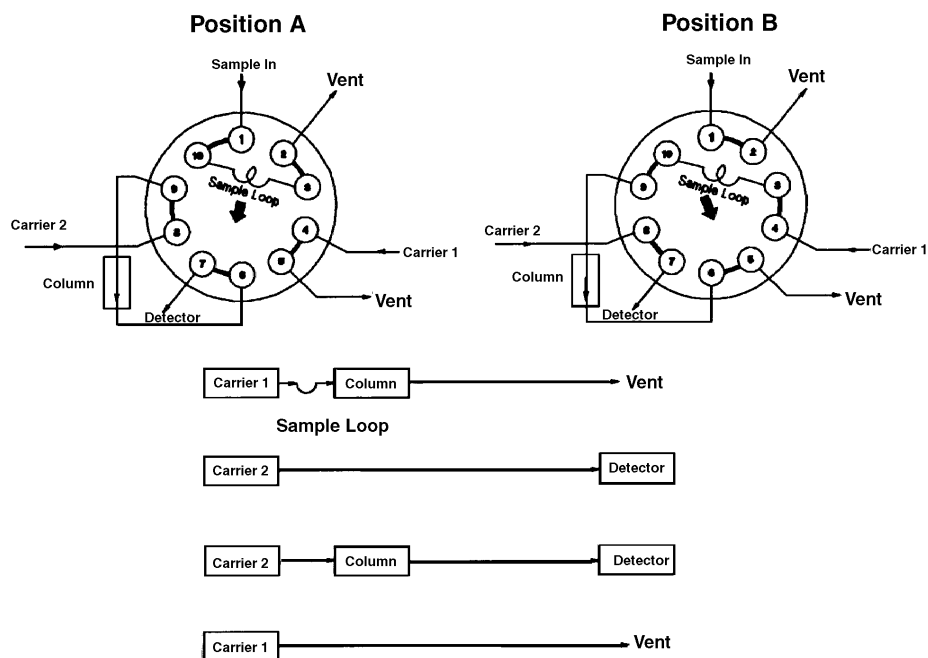


Figure 5
Valve Sampling System for the Analysis of Kr and N₂ in Grade 3.8

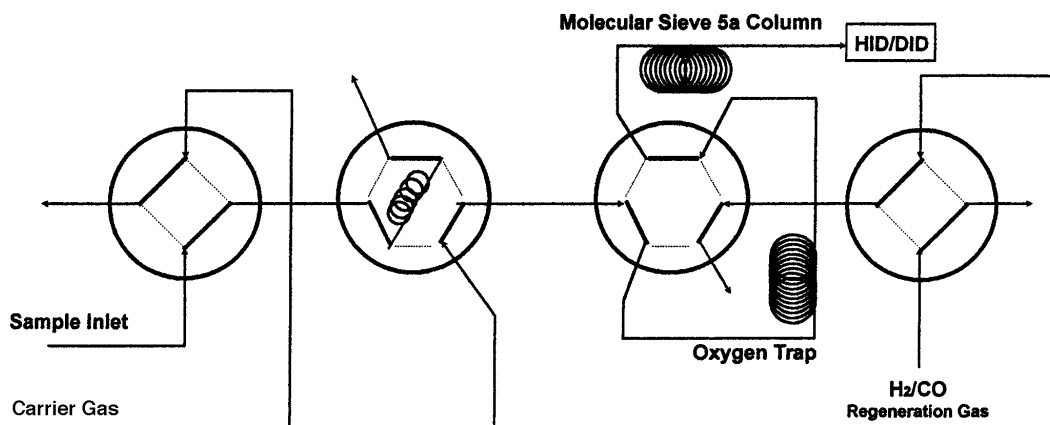


Figure 6
Analysis of Nitrogen and Argon in Oxygen Using Oxygen Trap in Grade 5.8



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SEMI C55-1104

SPECIFICATION FOR LIQUID CARBON DIOXIDE (CO₂) USED IN NEAR CRITICAL, CRITICAL AND SUPERCRITICAL APPLICATIONS, ≥ 99.99% 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 11, 2004. Initially available at www.semi.org September 2004; to be published November 2004.

1 Purpose

1.1 The purpose of this document is to provide a specification for liquid carbon dioxide (CO₂) that is used in near critical, critical and supercritical applications in the semiconductor industry.

2 Scope

2.1 This document provides purity requirements for all grades of liquid carbon dioxide that are used in near critical, critical and supercritical applications in the semiconductor industry.

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

NOTE 1: Minimum purity requirements. Some applications may require higher purity.

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 to determine the applicability of regulatory or other limitations prior to use.

3 Description/Definition

3.1 Carbon dioxide is a non-flammable, colorless, odorless compound that is stable at ambient conditions (25°C, 1 bar). Supercritical carbon dioxide exists at a temperature and pressure that exceeds the critical point of carbon dioxide. Near critical carbon dioxide exists in the liquid phase at a pressure that meets or exceeds the critical pressure and a temperature that is below its critical temperature but above a reduced temperature (absolute temperature divided by absolute critical temperature) of approximately 0.75.

4 Terminology

4.1 None.

5 Specifications

Table 1 Quality: ≥99.99%

<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Water	20 ppmv
Total Hydrocarbons expressed as Methane (THC)	50 ppmv
Particles	#1
Soluble and Insoluble Compounds Containing Metals (Na, Fe, Ni, Cu, Cr, Co, Ca, Mn, W, Mo) (each)	0.1 ppmw

#1: To be determined between supplier and user.

6 Referenced Standards

6.1 SEMI Standards

SEMI C10 — Guide for Determination of Method Detection Limits

SEMI C15 — Test Method for ppm and ppb Humidity Standards

SEMI F33 — Method for Calibration of Atmospheric Pressure Ionization Mass Spectrometer (APIMS)

6.2 ASTM Standard¹

E 1747-95(2000) — Guide for Purity of Carbon Dioxide Used in Supercritical Fluid Applications

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

¹ American Society for Testing and Materials International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555, Website: www.astm.org.

7 Physical Constants

7.1 The physical constants of carbon dioxide are given in Table 2 (for information only).

Table 2 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular Weight	44.01	44.01
Sublimation Point at 1 atm (14.7 psia)	-78.5°C	-109.3°F
Triple Point Temperature	-56.6°C	-69.9°F
Triple Point Pressure	5.1 atm	75.1 psia
Critical Temperature	31°C	87.8°F
Minimum Near Critical Temperature	-45.0°C	-49.1°F
Critical Pressure	73 atm	1070 psia
Density of gas at 21.1°C (70°F) and 1 atm	1.823 kg/m ³	0.1138 lb/ft ³
Density of Saturated Liquid at 25°C	713 kg/m ³	44.5 lb/ft ³

8 Analytical Procedures

8.1 *Water* — This procedure is for the determination of trace moisture (water) in carbon dioxide using an analyzer, such as a continuous flowing phosphorus pentoxide hygrometer.

8.1.1 *Detection Limit* — 100 ppbv or better

8.1.2 *Instrument Parameters*

8.1.2.1 *Flow Requirements* — Use appropriate flow, as recommended by manufacturer.

8.1.3 *Calibration Standard* — The analyzer shall be calibrated by reference to a standard gas calibration mixture containing a moisture content close to the expected value of the gas under test, in accordance with the instrument manufacturer's instructions. This calibration standard may be prepared using the procedure described in SEMI F33, Sections 13.2.2 and 13.2.3 and tested using the procedure described in SEMI C15.

8.1.4 Sample System

8.1.4.1 The sample line and associated valves must be clean and moisture free. They must be constructed from high cleanliness materials, such as electropolished stainless steel. The sample line should be purged dry if exposed to ambient moisture. The baseline moisture level should be less than 0.1 ppm.

8.1.4.2 Draw a continuous liquid sample from the source. The sample line should be insulated so that the sample does not boil due to heat infiltration. The distance between the sample point and the analyzer should be as small as possible to minimize boil-off.

8.1.4.3 Heat the sample so that it is completely vaporized, leaving absolutely no liquid or solid carbon dioxide or water residue.

8.1.4.4 Maintain the sample temperature at the analyzer inlet above its condensation point. A sample temperature of 40°C is recommended.

8.1.4.5 Figure 1 illustrates one potential sampling configuration.

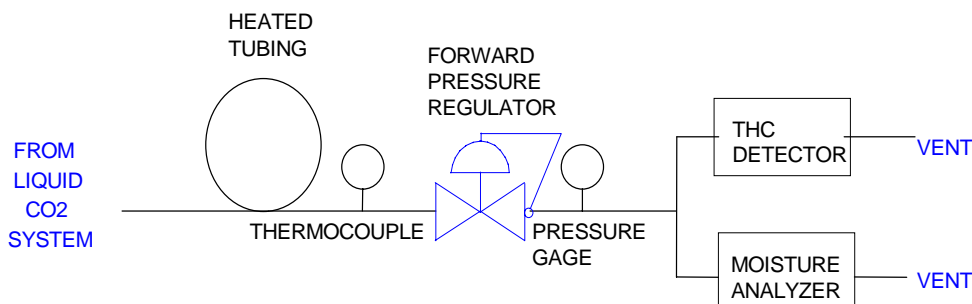


Figure 1
Potential Hydrocarbon and Water Sampling System

8.1.5 Operating Procedure

8.1.5.1 Follow the procedures specified in the instrument manufacturer's manual.

8.1.5.2 Allow the system to run until a stable reading is obtained for 20 minutes.

8.1.5.3 Read and record the concentration of moisture indicated on the moisture analyzer, in ppmv. The result obtained shall not exceed the value specified in Section 5 of this specification.

8.2 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons expressed as methane in carbon dioxide using a total hydrocarbon analyzer.

8.2.1 *Detection Limit* — 100 ppbv or better.

8.2.2 *Instrument Parameters*

8.2.2.1 *Flow Requirements* — Use appropriate flow, as recommended by manufacturer.

8.2.3 *Calibration Standard* — 1 ppmv methane in nitrogen.

8.2.4 *Sampling System*

8.2.4.1 The sample line and associated valves must be clean and hydrocarbon free. They must be constructed from high cleanliness materials, such as electropolished stainless steel. The sample line should be cleaned if exposed to hydrocarbons, such as oils or greases.

8.2.4.2 Draw a continuous liquid sample from the source. The sample line should be insulated so that the sample does not boil due to heat infiltration. The distance between the sample point and the analyzer should be as small as possible to minimize boil-off.

8.2.4.3 Heat the sample so that it is completely vaporized, leaving absolutely no liquid or solid carbon dioxide or water residue. Note that if detection of heavy hydrocarbons (>C10) is important, it may not be feasible to completely vaporize the sample. Therefore, a concentrator method, such as that described in ASTM E 1747, Section 6, may be required.

8.2.4.4 Maintain the sample temperature at the analyzer inlet above its condensation point. A sample temperature of 40°C is recommended.

8.2.4.5 Figure 1 illustrates one potential sampling configuration.

8.2.5 Operating Procedure

8.2.5.1 Follow the procedures specified in the instrument manufacturer's manual.

8.2.5.2 Allow the system to run until a stable reading is obtained for 20 minutes.

8.2.5.3 Read and record the concentration of hydrocarbons as methane indicated on the total hydrocarbon analyzer, in ppmv. The result obtained shall not exceed the value specified in Section 5 of this specification.

8.3 *Metals* — This procedure is for the determination of insoluble and soluble metals in carbon dioxide using inductively coupled plasma-mass spectroscopy (ICP-MS), ICP-AES. Specific components to be monitored are Na, Fe, Ni, Cu, Cr, Co, Mn, Ca, Mo and W.

NOTE 2: The specified method does not detect volatile metals.

8.3.1 *Detection Limit* — Determined per SEMI C10.

8.3.2 *Instrument Parameters*

8.3.3 *Calibration Standards* — Consult with instrument manufacturer instructions. All instrument calibration standards should be prepared from NIST-traceable reference standards.

8.3.4 *Sampling System*

8.3.4.1 Two sample preparation methods are approved for metals analysis. These are a residue method and a filtration method. The user may select between these techniques based on convenience, economic considerations, or other situation specific conditions.

8.3.4.1.1 *Residue Method* — A CO₂ sample is transferred to an ambient pressure vessel and allowed to sublime. The remaining metal residue is dissolved in nitric acid.

8.3.4.1.1.1 The sample line and associated valves must be clean and metal particle free. They must be constructed from high cleanliness materials, such as electropolished stainless steel.

8.3.4.1.1.2 The sample system must be purged with high-purity nitrogen prior to and after sampling. Prior to sampling, the entire sampling system should be purged by allowing high purity nitrogen to flow through the system at 5–10 psig for a minimum of three minutes.

8.3.4.1.1.3 When the purge is complete, draw a sufficient mass of liquid carbon dioxide sample from the source to obtain a meaningful metals level. A valve should be installed in the sample line to flash the liquid sample from the operating pressure to near ambient pressure. This will cause the sample to form a solid/vapor mixture. This mixture should then be directed to the bottom of a vented sample flask. The sample flask should have a volume of 100 ml and should be composed of polypropylene or other suitable materials. It should be cleaned prior to use using DI water.

8.3.4.1.1.4 Solid CO₂ and any metals will deposit in the sample flask. When sampling is complete, the resulting solid CO₂ should be allowed to warm to ambient temperature and sublime. As a result, vapor CO₂ will leave the sample flask.

8.3.4.1.1.5 After all of the CO₂ has been vented from the sample flask, the system should be purged with nitrogen for 15 minutes.

8.3.4.1.1.6 The sample flask should then be filled with a solution of 5% ultrapure HNO₃/DI H₂O. This solution may be spiked with an appropriate internal standard. Allow the metal residue contained in the flask to dissolve in the solution for a minimum of one hour. Note that a different solution may be used, depending on possible interferences with a given analytical technique. The solution contained in sample flask is ready for injection into an ICP-MS.

8.3.4.1.1.7 Figure 2 illustrates a potential configuration for this sampling system.

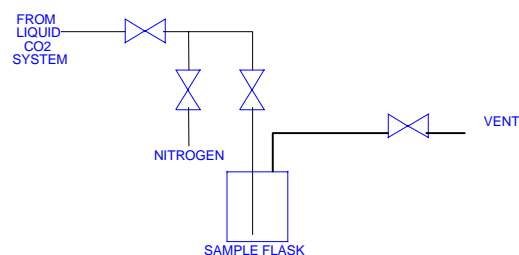


Figure 2
**Potential Metals Sample Preparation System-
Residue Method**

8.3.4.1.2 *Filtration Method* — A Teflon® filter is used to remove metallic impurities from CO₂. The collected metallic impurities are dissolved in nitric acid.

8.3.4.1.2.1 The sample line and associated valves must be clean and metal particle free. They must be constructed from high cleanliness materials, such as electropolished stainless steel.

8.3.4.1.2.2 A sampling manifold, such as that depicted in Figure 3 may be employed. Referring to this figure, V1, V2, V3, V4, V5 are shut off valves, MV is a metering valve, PSV is a pressure relief device set to 2000 psi and FH is a filter holder that accommodates a 0.2 µm Teflon® filter. A flow meter at the exit end of the sampling system is used to measure the sample flow. A heating device such as a heat lamp is used, downstream of the filter to totally vaporize the liquid CO₂ so that an accurate flow measurement can be made.

8.3.4.1.2.3 The sample system must be purged with high-purity nitrogen prior to sampling. Close valves V1, V2, V3, V4, V5 and MV. Open V2, V3, V4, V5 and MV to establish a flow of nitrogen that is filtered to remove particles smaller than 0.2 micron. Allow nitrogen to flow through the system for a minimum of 3 minutes. Close V2, V3, V4, and V5.

8.3.4.1.2.4 Clean a 0.2 µm Teflon® filter using a 10% by weight HNO₃ solution. Install the filter in the filter holder FH. Perform this operation in a Class 100 laminar flow hood.

8.3.4.1.2.5 Install the filter holder in the sampling system.

8.3.4.1.2.6 Open V1 and V3. Allow CO₂ to flow until liquid is observed at the flow meter.

8.3.4.1.2.7 Turn on the heating device.

8.3.4.1.2.8 Close V1, then simultaneously open V4 and V5 to avoid pressurizing the filter from either end. Slowly open V1. Close V3 to establish CO₂ flow through the filter. Accurately note the flow meter reading and the initial sampling time.

8.3.4.1.2.9 Allow a sufficient volume of CO₂ to flow through the filter to collect a metals sample that will produce a meaningful reading on the ICP-MS. This volume will be a function of the metals level present and must be estimated.

8.3.4.1.2.10 Close V1 and V4, noting the final sample time. Based on the flow meter reading and initial and final sample times, calculate the total amount of CO₂ sampled.

8.3.4.1.2.11 Purge CO₂ from the sampling system by opening V2 and V3, allowing nitrogen to flow. When the flow meter reading becomes very low or stops, close V4 and V5.

8.3.4.1.2.12 Remove the filter holder from the sampling manifold and take it to a clean room for disassembly. V2 should be opened to purge during disconnection.

8.3.4.1.2.13 Disassemble the filter holder inside a Class 100 laminar flow hood. Remove the Teflon® filter, using Teflon® tweezers. Put the filter in a PFA bottle containing a 10% by weight HNO₃ solution.

8.3.4.1.2.14 Place the PFA bottle containing the filter in an ultrasonic bath for 30 minutes. This will help to dislodge any particles that are trapped on the filter and assist in their dissolution in the HNO₃ solution. The solution contained in the PFA bottle is ready for injection into an ICP-MS.

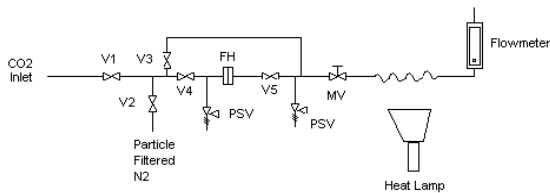


Figure 3
Potential Metals Sample Preparation System-
Filtration Method

8.3.5 Operating Procedure

8.3.5.1 Follow the procedures specified in the instrument manufacturer's manual for operating the ICP-MS and introducing the sample solution.

8.3.5.2 Read and record the concentration of each relevant metal, in ppmw. The result obtained shall not exceed the value specified in Section 5 of this specification.

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SEMI C56-0305

SPECIFICATIONS AND GUIDELINES FOR DICHLOROSILANE (SiH₂Cl₂)

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 November 4, 2004. Initially available at www.semi.org January 2005; to be published March 2005.

NOTICE: This document replaced C3.18 and C3.31 in 2005.

1 Purpose

1.1 The purpose of this document is to provide a series of specifications for different grades of dichlorosilane (SiH₂Cl₂) that are used in the semiconductor industry.

2 Scope

2.1 This document covers requirements for all grades of dichlorosilane (SiH₂Cl₂) used in the semiconductor industry.

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

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 Description

3.1 Dichlorosilane is a flammable, corrosive, colorless liquid which hydrolyzes in the presence of moisture. It oxidizes readily and rapidly to release hydrogen chloride. It is easily ignited in the presence of air.

4 Referenced Standards

4.1 SEMI Standards

SEMI C1 — Specifications for Reagents

SEMI C3 — Specifications for Gases

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

5 Terminology

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

6 Requirements

6.1 Purity and other requirements for the various grades of dichlorosilane are given in Table 1.

Table 1 Impurity and Other Requirements for Various Grades of Dichlorosilane

<i>Previous SEMI Reference #</i>	<i>C3.18-1102 (Specification)</i>	<i>C3.31-1102 (Specification)</i>
Purity	97%	99%
Impurities	Maximum Acceptable Level ^{#1}	
All Other Chlorosilanes (Monochlorosilane, Silicon Tetrachloride, Trichlorosilane)	3% (liquid phase)	1% (liquid phase) ^{#2}
Aluminum	1.0 ppbw (vapor phase derived)	1.0 ppbw (vapor phase derived)
Arsenic	0.5 ppbw (vapor phase derived)	0.2 ppbw (vapor phase derived)
Boron	0.3 ppbw (vapor phase derived)	0.1 ppbw (vapor phase derived)
Carbon	10 ppmw (vapor phase derived)	1 ppmw (vapor phase derived)
Iron	50 ppbw (liquid phase)	50 ppbw (liquid phase)
Phosphorus	0.3 ppbw (vapor phase derived)	0.3 ppbw (vapor phase derived)

^{#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.

^{#2} Monochlorosilane not to exceed 0.5%

^{#3} This specification applies to the appropriate phase of the cylinder as delivered.

^{#4} The liquid phase purity is to be based on the chlorosilane determination. In addition, argon + helium are to be controlled in the vapor phase to less than 0.5%. This argon + helium determination will not affect the percent purity determination.

^{#5} Initial purging of the vapor phase is recommended to reduce inert gas content used in the cylinder fill procedure.

^{#6} Analytical procedures for the doping elements, metals and selected impurities are to be determined between the user and supplier at the present time.

7 Electrical Requirements

7.1 Electrical requirements for the various grades of dichlorosilane are given in Table 2

Table 2 Electrical Requirements for Various Grades of Dichlorosilane

<i>Previous SEMI Reference #</i>	<i>C3.18-1102 (Specification)</i>	<i>C3.31-1102 (Specification)</i>
Resistivity ^{#1}	Greater than 50 ohm-cm (n-type) ^{#2}	Greater than 150 ohm-cm (n-type) ^{#2}

^{#1} The resistivity measurement technique is to be determined between supplier and user.

^{#2} Monocrystalline silicon formed from this material, not the material itself.

8 Physical Constants

8.1 The physical constants of dichlorosilane are given in Table 3 (for information only).

Table 3 Physical Constants of Dichlorosilane (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	100.99	100.99
Boiling point at 1 atm	8.2°C	46.8°F
Density of gas at 25°C (77°F) and 1 atm	4.168 kg/m ³	0.260 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	3.48	3.48
Density of liquid at 25°C (77°F)	122 kg/m ³	7.62 lb/ft ³

9 Analytical Procedures for Purity of 97% of Dichlorosilane

NOTE 1: The sample is extremely flammable and corrosive. Test for leaks before sampling. Ensure good electrical grounding.

9.1 *Other Chlorosilanes* — This procedure is for the determination of other chlorosilanes (monochlorosilane, trichlorosilane and silicon tetrachloride) in dichlorosilane using a gas chromatograph with a thermal conductivity detector.

9.1.1 *Detection Limit* — 10 ppm.

9.1.2 *Instrument Parameters*

9.1.2.1 *Column*: 3.05 m (10 ft) by 3.2 mm (1/8 in) stainless steel tubing packed with 20% OV-101 on 80/100 Chromosorb W-HP or equivalent.

9.1.2.2 *Carrier Flow*: 20 mL/min helium.

9.1.2.3 *Sample Volume*: 0.5 to 1.0 mL.

9.1.2.4 *Temperatures*:

Detector	350°C
Column	60°C ramped to 325°C at 10°C/min
Inlet	300°C

9.1.3 *Operating Procedure*

9.1.3.1 Inject a known sample of monochlorosilane, trichlorosilane and silicon tetrachloride in dichlorosilane into the column using a gas sampling valve. Record the retention times and peak areas. The order of elution is monochlorosilane, dichlorosilane, trichlorosilane, silicon tetrachloride.

9.1.3.2 Inject the sample to be tested in the same manner as in ¶9.1.3.1. Record the areas of the peaks which have the same retention times as those in ¶9.1.3.1.

9.1.3.3 Repeat ¶9.1.3.1.

9.1.3.4 Calculate the purity of dichlorosilane in the sample, using the equations stated below. The result may not exceed the specification in Table 1 of this standard.

9.1.4 *Calculations*

9.1.4.1 Determine the total area of the chlorosilane peaks (A_T) by adding the areas of the four peaks.

9.1.4.2 Divide the area of the dichlorosilane peak by A_T . Multiply the result by 100% to obtain the purity of the dichlorosilane.

10 Analytical Procedures for Purity of 99% of Dichlorosilane (see Note 1 in §9)

10.1.1 *Other Chlorosilanes* — Follow the procedure in §9 except add the following calculation for monochlorosilane.

10.1.2 *Calculations for monochlorosilane*

10.1.2.1 Calculate the percentage the monochlorosilane in the sample using the equation stated below. The result may not exceed the specification in table 1 of this standard.

10.1.2.2 Divide the area of the monochlorosilane peak by A_T . Multiply the result by 100% to obtain the percentage of monochlorosilane.



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 C57-0305

SPECIFICATIONS AND GUIDELINES FOR ARGON

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 December 10, 2005. Initially available at www.semi.org January 2005; to be published March 2005.

NOTICE: This document replaced SEMI C3.1, SEMI C3.42, and SEMI C3.46 in 2005.

1 Purpose

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

2 Scope

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

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

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 Description

3.1 Argon is the most abundant member of the rare gas family. It is monatomic and is characterized by its extreme chemical inactivity. Argon is a colorless, odorless and tasteless gas somewhat soluble in water. It is normally delivered and stored as a cryogenic liquid.

4 Limitations

4.1 None.

5 Referenced Standards

5.1 SEMI Standards

SEMI C1 — Specifications for Reagents

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 Argon are given in Table 1.

Table 1 Impurity and Other Requirements for Various Grades of Argon

<i>Previous SEMI Reference #</i>	<i>C3.1-1101 (Specification)</i>	<i>C3.46-1102 (Specification)</i>	<i>C3.42-90 (Guideline)</i>
Grade	4.8	5.2	6.0
Purity	99.998%	99.9992%	99.99990% ^{#1}
Impurities	Maximum Acceptable Level (ppm) ^{#2}		
Carbon monoxide and carbon dioxide (CO + CO ₂)	0.5	0.5	N/A
Carbon Dioxide (CO ₂)	N/A	N/A	0.05
Carbon Monoxide (CO)	N/A	N/A	0.05
Hydrogen (H ₂)	1	1	0.10
Nitrogen (N ₂)	10	5	0.5
Oxygen (O ₂)	2	0.5	0.05
Total Hydrocarbons expressed as Methane (THC)	0.5	0.5	0.1
Water (H ₂ O) (v/v)	1	0.5	0.1
TOTAL SPECIFIED IMPURITIES	15	8	0.95
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 argon are given in Table 2 (for information only).

Table 2 Physical Constants of Argon (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	39.948	39.948
Boiling point at 1 atm	−185.9°C	−302.6°F
Density of gas at 21.1°C (70°F) and 1 atm	1.656 kg/m ³	0.1034 lb/ft ³
Specific gravity of gas at 21.1°C and 1 atm (air = 1)	1.38	1.38
Density of liquid at boiling point	139 kg/m ³	8.698 lb/ft ³

9 Analytical Procedures for Grade 4.8 Argon

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

NOTE 2: All gases used in the analysis of the sample should contain no more than 10% of the sample value of the component of interest unless otherwise specified.

9.1 *Carbon Monoxide and Carbon Dioxide* — This procedure is for the determination of carbon monoxide and carbon dioxide in argon using a gas chromatograph with a flame ionization detector and methanizer.

9.1.1 *Detection Limit* — 100 ppb

9.1.2 *Instrument Parameters*

9.1.2.1 *Column*: Porapak T or Q, 3 m (9.8 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel; or Chromosorb 102, 2 m (6.6 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel; or equivalent.

9.1.2.2 *Carrier Flow*: 30 mL/min helium



9.1.2.3 *Sample Volume:* 0.5 to 2.0 mL

9.1.2.4 *Temperatures:*

Detector	280°C
Column Oven	60°C
Methanizer	350°C

9.1.3 *Calibration Standards* — 1–5 ppm carbon monoxide, 1–5 ppm carbon dioxide, balance argon.

9.1.4 *Operating Procedure*

9.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas. The order of elution is carbon monoxide, carbon dioxide.

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

9.1.4.3 Repeat ¶9.1.4.1.

9.1.4.4 Compare the average peak areas of the calibration standard to that of the argon sample being tested. Calculate the concentrations of carbon monoxide and carbon dioxide, using the formula below.

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

9.1.4.5 The result may not exceed the specification in Table 1.

9.2 *Hydrogen and Nitrogen* — This procedure is for the determination of hydrogen and nitrogen in argon using a gas chromatograph with a helium ionization detector.

9.2.1 *Detection Limit* — 0.5 ppm

9.2.2 *Instrument Parameters*

9.2.2.1 *Column:* 5A molecular sieve, 65.6 m (20 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel or equivalent.

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

9.2.2.3 *Sample Volume:* 1.0 to 3.0 mL

9.2.2.4 *Temperatures:*

Detector	125°C
Column Oven	25°C

9.2.3 *Calibration Standard* — 1–5 ppm hydrogen in argon, 10–30 ppm nitrogen in argon.

9.2.4 *Operating Procedure*

9.2.4.1 Inject the calibration standard into the column using a gas sampling valve. Record the retention times and peak areas.

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

9.2.4.3 Repeat ¶9.2.4.1. (See Note 1.)



9.2.4.4 Compare the average peak area of the calibration standard to that of the argon sample being tested. Calculate the concentration of hydrogen and nitrogen, using the formula below.

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

9.2.4.5 The result may not exceed the specifications in Table 1.

9.3 *Oxygen* — This procedure is for the determination of oxygen in argon using a continuous flow analyzer using an electrochemical method.

9.3.1 *Detection Limit* — 100 ppb.

9.3.2 *Flow Rate* — Set sample flow rates in accordance with the instrument manufacturer's instructions.

9.3.3 *Calibration Standard* — 3–15 ppm oxygen in argon or in accordance with the instrument manufacturer's instructions.

9.3.4 *Operating Procedure*

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

9.3.4.2 Introduce argon sample and record oxygen reading. The result may not exceed the specification in Table 1.

9.4 *Total Hydrocarbons* — This procedure is for the determination of total hydrocarbons in argon using a continuous flow flame ionization detector equipped total hydrocarbon analyzer.

NOTE 3: The 0–1 range can be used provided that zero and span gas standards in hydrogen with known levels of hydrocarbons between 0 and 1 ppm are used in the calibration of the analyzer.

NOTE 4: As the flow rate and heat capacity of the matrix gas affect the instrument output, the zero gas matrices should be coincided with that of the sample gas.

NOTE 5: The effective response of a flame ionization detector-equipped total hydrocarbon analyzer to different hydrocarbons can vary and should be approximated. However, the response of the common hydrocarbon impurities in argon can be accurately totaled and compared to methane.

9.4.1 *Detection Limit* — 0.1 ppm.

9.4.2 *Flow Requirements*

9.4.2.1 High purity, hydrocarbon-free (less than 0.1 ppm) hydrogen: 35–40 mL/min or 40% hydrogen in either helium or nitrogen matrix at 75–80 mL/min.

9.4.2.2 Dry, hydrocarbon-free (less than 0.1 ppm) air: 350–400 mL/min.

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

9.4.3 *Calibration Standards*

9.4.3.1 Zero argon with known quantity of hydrocarbons at 0.1 ppm level.

9.4.3.2 The upper level span gas not exceeding five times the concentration of the specification.

9.4.4 *Operating Procedure*

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

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

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

9.4.4.4 Introduce argon sample into the analyzer and read the quantity of hydrocarbons on the analyzer meter. The result may not exceed the specification in Table 1.

9.5 *Water* — This procedure is for the determination of trace moisture (water) in argon using a continuous flowing piezoelectric hygrometer.

NOTE 6: The sampling system and hygrometer should 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 7: Other hygrometers may also be used, e.g. CRDS, FTIR, TDLAS, and vibrating quarts.

9.5.1 *Detection Limit* — 0.1 ppm (vol/vol) at -90°C (-130°F).

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

9.5.3 *Calibration Standards* — Construct a calibration curve which contains at least three points covering the range of interest. Verify the standards employed independently by another analytical method.

9.5.4 *Operating Procedure*

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

9.5.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and the piezoelectric moisture hygrometer until a stable reading is obtained.

9.5.4.3 Determine the moisture content of the argon sample by comparing the reading to calibration curve. The result may not exceed the specification in Table 1.

10 Analytical Procedures for Grade 5.2 Argon (See Notes 1 and 2 in §9)

10.1 *Carbon Monoxide and Carbon Dioxide* — Use the procedure in ¶9.1 for the analysis of carbon monoxide and carbon dioxide except that the calibration standard should be 0.2–1 ppm carbon monoxide, 0.2–1 ppm carbon dioxide with balance Ar.

10.2 *Hydrogen and Nitrogen* — Use the procedure in ¶9.2 for the analysis of hydrogen and nitrogen except that the calibration standard should be 5–25 ppm nitrogen in argon.

10.3 *Oxygen* — Use the procedure in ¶9.3 for the analysis of oxygen except that the calibration standard should be 0.5–2.5 ppm oxygen in argon.

10.4 *Total Hydrocarbons* — Use the procedure in ¶9.4 for the determination of total hydrocarbons.

10.5 *Water* — Use the procedure in ¶9.5 for the determination of water.

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SEMI C58-0305

SPECIFICATIONS AND GUIDELINES FOR HYDROGEN

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 December 10, 2004. Initially available at www.semi.org February 2005; to be published March 2005.

1 Purpose

1.1 The purpose of this document is to provide a series of specifications for different grades of Hydrogen (H_2) that are used in the semiconductor industry.

2 Scope

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

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

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 Description

3.1 Hydrogen is a colorless gas. Its vapors are highly flammable, colorless, tasteless, and nontoxic. It is the lightest gas known.

4 Limitations

4.1 None.

5 Referenced Standards

5.1 *SEMI Standards*

SEMI C1 — Specifications for Reagents

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 hydrogen are given in Table 1.