



SEMI C3.58-0303

SPECIFICATION FOR OCTAFLUOROCYCLOBUTANE, C₄F₈, ELECTRONIC GRADE IN 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 October 25, 2002. Initially available at www.semi.org December 2002; to be published March 0303. Originally published June 2000.

NOTICE: This entire document was completely revised in 2003.

1 Description

1.1 Octafluorocyclobutane, also known as perfluorocyclobutane, is a gas under normal atmospheric conditions (15°C and 760 mm Hg).

1.2 The gas is a non-flammable, non-toxic, colorless and odorless gas which is stable under normal conditions.

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.

2 Specifications

Purity: 99.999%

Impurities	Maximum Acceptable Level
Oxygen	1 ppmv
Moisture	3 ppmv
Other organic (PFC-1216, PFC-31-10mc, PFC-1318my, PFC-31- 10my, CFC-114, CFC-114a, THF)	10 ppmv
Acidity (as HCl)	100 ppbw
Air (N ₂ , O ₂ , CO, CO ₂)	3 ppmv

3 Referenced Standards

None.

4 Physical Constants (for information only)

Formula	C ₄ F ₈ or CF ₂ • CF ₂ • CF ₂ • CF ₂
Molecular weight	200
Melting point	-41.4°C
Boiling point	-6°C
Relative density, gas	6.9 (air = 1)
Relative density, liquid	1.6 (water = 1)
Vapor pressure 20°C	2.7 bar

Appearance/Color	Colorless gas
Odor	none

5 Analytical Procedures

NOTE 1: Samples of octafluorocyclobutane should be equilibrated at no less than 30°C (85°F) for at least 24 hours prior to analysis to ensure adequate vapor pressure for analysis and consistency.

5.1 *Acidity* — This procedure is for the determination of acidity in octafluorocyclobutane utilizing an aqueous conductivity measurement. Calculations are made relating sample conductivity to free acidity, calculated as hydrochloric acid, in the sample.

5.1.1 *Detection Limit* — 30 ppb as HCl.

5.1.2 *Apparatus*: A conductivity bridge and cell, heat gun, graduated cylinders, gas dispersion tube, wide-mouth polyethylene bottle, hypodermic syringes, polyethylene beaker, water purification system.

5.1.3 *Reagents*

5.1.3.1 Hydrochloric acid, 0.01 N standard in SDA-2B ethyl alcohol (store the standard in separate 50-mL serum capped vials).

5.1.3.2 CFC-11, special (analyzed to have an aqueous conductance of < 0.5 µmho/cm). If CFC-11 is not available, a similar degreasing solvent can be substituted, such as HFC-43-10.

5.1.3.3 Distilled and de-ionized water, aqueous conductivity < 0.5 µmho/cm, prepared immediately prior to use by passing distilled water through a water purification system and analyzing it with a conductivity bridge. DO NOT STORE DE-IONIZED WATER FOR USE IN THIS ANALYSIS.

5.1.3.4 RBS-35 cleaner.

5.1.4 *Operating Procedure*

5.1.4.1 Attach a suitable regulator to the cylinder outlet.



5.1.4.2 *Conductivity bridge/cell* — Store the conductivity cell in a polyethylene beaker containing de-ionized water when not in use.

5.1.4.3 *Calibration*

5.1.4.3.1 Add CFC-11 to the 20 mL calibration mark of the polyethylene (PE) bottle. Immediately cap the bottle and shake contents vigorously for approximately 30 seconds. Discard the CFC-11.

5.1.4.3.2 Fill the bottle again with CFC-11 to the 20 mL mark. Immediately add 100 mL of de-ionized (DI) water to the bottle, cap and shake vigorously for approximately 120 seconds.

5.1.4.3.3 Rinse the conductivity cell with DI water and shake it dry. Uncap the PE bottle and dip the conductivity cell into the water layer (top layer). Read and record the conductivity value. This reading will serve as the "BLANK" value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water. Immediately cap the PE bottle.

5.1.4.3.4 Fill and flush a clean, dry, 50 microliter syringe several times with the 0.01 N standard, hydrochloric acid in SDA-2B ethanol. Fill the syringe to a volume of 30 microliters. Holding the syringe vertically with needle pointing up, tap the barrel gently to permit air bubbles to rise to the top. Slowly depress the plunger until the plunger tip is aligned exactly with the 20 microliter mark. Immediately uncap the PE bottle and inject the syringe contents into the bottle. Immediately cap the bottle. Shake the bottle vigorously for approximately 120 seconds. Uncap the PE bottle and dip the conductivity cell into the water layer. Read and record the conductivity value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water.

5.1.4.3.5 Calculate the nanograms (ng) of hydrochloric acid used in the standard from the following equation:

$$\text{ng HCl} = \frac{V \times N \times \text{MEQ} \times 10^9}{10^3}$$

Where: ng HCl = nanograms of HCl in the standard

V = HCl volume, microliters

N = HCl normality

MEQ = HCl milliequivalent weight
(g/meq) = 0.036461

5.1.4.3.6 Calculate the calibration factor as follows:

$$\text{ng HCl}/\mu\text{mho} = \frac{A}{(C2-C1)}$$

Where: ng HCl/ μmho = calibration factor

A = ng HCl in standard

C1 = blank conductivity, μmho

C2 = standard conductivity, μmho

5.1.4.3.7 Repeat procedure at least six times. Use the average of the six calibration factors for sample calculations.

5.1.4.4 *Sampling and Analysis*

5.1.4.4.1 Add CFC-11 or a similar degreasing solvent to the 20 mL calibration mark of the polyethylene (PE) bottle. Immediately cap the bottle and shake contents vigorously for approximately 30 seconds. Discard the CFC-11.

5.1.4.4.2 Fill the bottle again with CFC-11 to the 20 mL mark. Immediately add 100 mL of DI water to the bottle, cap and shake vigorously for approximately 120 seconds.

5.1.4.4.3 Rinse the conductivity cell with DI water and shake it dry. Uncap the PE bottle and dip the conductivity cell into the water layer (top layer). Read and record the conductivity value. This reading will serve as the "BLANK" value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water. Immediately cap the PE bottle.

5.1.4.5 Weigh the sample cylinder and record. Attach PE gas dispersion tube to the sample cylinder valve. Uncap the PE bottle and lower the gas dispersion tube into the water. Open the cylinder valve and purge sample through the bottle such that about 150 g of sample are added to the bottle over a 60 minute period. Close the cylinder valve and immediately remove the gas dispersion tube and cap the bottle. Record the final weight of the capped sample cylinder.

5.1.4.6 Shake the PE bottle vigorously for approximately 120 seconds. Read and record the conductivity value as done previously.

5.1.4.6.1 Calculate the sample's free acidity using the following equation:

$$\text{ppb HCl} = \frac{(C_2 - C_1) \times F}{W}$$

Where: ppb HCl = free acidity

C₁ = blank conductivity, $\mu\text{mho}/\text{cm}$
 C₂ = sample conductivity, $\mu\text{mho}/\text{cm}$
 F = calibration factor, ng HCl/ μmho
 W = sample weight used in analysis, grams

6 Analytical Procedures - Instrumental Analysis

6.1 Air (N_2, O_2 , CO, CO_2) and Oxygen — This procedure is for the determination of air components in octafluorocyclobutane using a gas chromatograph with a discharge ionization detector. Column switching is used to separate and elute the components and to backflush octafluorocyclobutane. One method/column is used for N_2 , O_2 , and CO and a second method/column is used for CO_2 . The sample should be taken from the vapor in the container directly into the instrument sampling system. The container being analyzed should have equilibrated at approximately 30°C before analysis is begun.

6.1.1 Detection Limits — Nitrogen, 10 ppb vol; Oxygen, 50 ppb vol; Carbon monoxide, 10 ppb vol; Carbon dioxide, 10 ppb vol.

6.1.2 Instrument Parameters

6.1.2.1 Columns: Precolumn: Haysep DB on Carbo pack B, 80/100 mesh, 2 ft \times 1/8 in stainless steel. N_2, O_2, CO column: Molecular Sieve 5a, 80/100 mesh, 8 ft \times 1/8 in stainless steel. CO_2 column: Haysep DB on Carbo pack B, 80/100 mesh, 8 ft \times 1/8 in stainless steel.

6.1.2.2 Gas Flows: Helium carrier gas 40 mL/min for each column.

6.1.2.3 Sample volume: 1.0 mL

6.1.2.4 Temperatures: Detector setpoint = 25°C (actual is higher ~60°C due to proximity to the oven), Oven 77°C isothermal.

6.1.2.5 Calibration Standard: 1-30 ppm vol oxygen, nitrogen, carbon monoxide, and carbon dioxide in helium.

6.1.2.6 Chromatograms — See Figures 1,2.

6.2 Organic Impurities — This procedure is for the determination of organic impurities in

octafluorocyclobutane using a gas chromatograph with a packed column and flame ionization detector.

6.2.1 Detection Limits

PFC-1216 (hexafluoropropene),

PFC-31-10mc (n-decafluorobutane),

PFC-1318my (octafluorobutene, cis and trans)

0.1 vol, ppm

PFC-31-10my (iso-decafluorobutane),

CFC-114 (1,2-dichloro-1,1,2,2-tetrafluoroethane),

CFC-114a(1,1-dichloro-1,2,2,2-tetrafluoroethane), THF(tetrahydrofuran)

0.2 vol, ppm

6.2.2 Instrument Parameters

6.2.2.1 Column — 1% SP-1000, on Carbo pack B, 60/80 mesh, 24 ft \times 1/8 in stainless steel or equivalent.

6.2.2.2 Gas Flows — Helium carrier gas, 20 mL/minute.

6.2.2.3 Sample volume — 1.0 mL vapor.

6.2.2.4 Temperatures — Injector = 150°C, Detector = 250°C, Oven: 75°C for 10 minute, increase 8°C/min to 200°C and hold for 10 minutes.

6.2.2.5 Calibration Standard: 1-50 ppm vol, PFC-1216, PFC-31-10my, PFC-31-10mc, PFC-1318my (cis and trans), CFC-114/114a, and THF in octafluorocyclobutane.

6.2.2.6 Chromatogram — See Figure 3.

6.3 Water — This procedure is for the determination of trace moisture (water) in Octafluorocyclobutane, using oscillating crystal technology. The instrument monitors the change in vibrational frequency of a hygroscopically sensitized quartz crystal when it is exposed alternately to wet and dry sample gas. The change in vibrational frequency is a function of the amount of moisture sorbed from the wet sample gas. The sample should be taken from the vapor in the container directly into the instrument sampling system. The container being analyzed should have equilibrated at approximately 30°C before analysis is begun.

6.3.1 Detection Limit — 0.04 ppm vol (40 ppb vol).

6.3.2 Sample Pressure and Flow — These parameters should be set in accordance with instrument manufacturer's instructions.

6.3.3 Operation Check — The instrument should be checked periodically for correct operation. The instrument should be zeroed by flowing reference gas, which is dry octafluorocyclobutane (dried by use of an ultra dryer system provided by the instrument manufacturer). Then the instrument should be calibrated by use of the internal moisture generator (permeation tube) which has been certified by the manufacturer at some known moisture level. When not in use the instrument should be purged with dry gas (i.e., helium or nitrogen).

6.3.4 Operation Procedures

6.3.4.1 Initiate flow of the reference gas to the instrument and allow 30 minutes for stabilization.

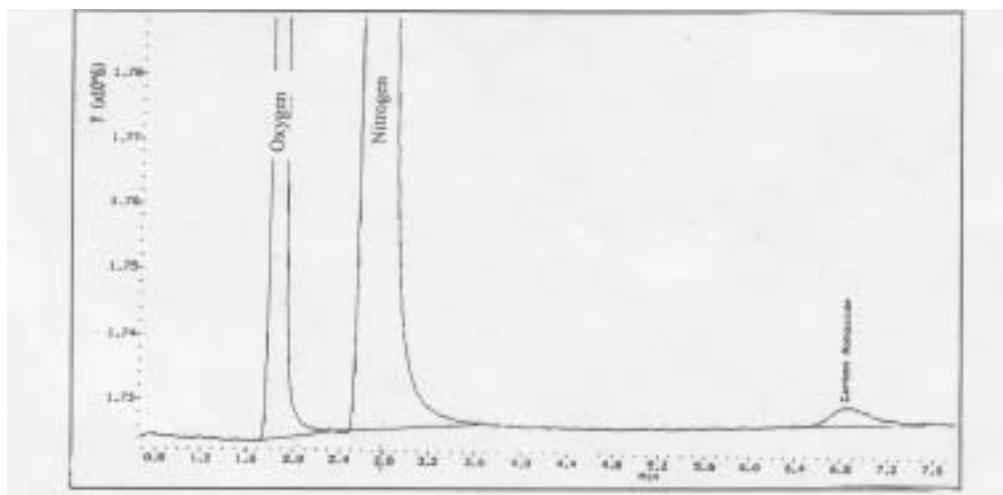


Figure 1
Nitrogen, Oxygen, and Carbon Monoxide Determination in Octafluorocyclobutane

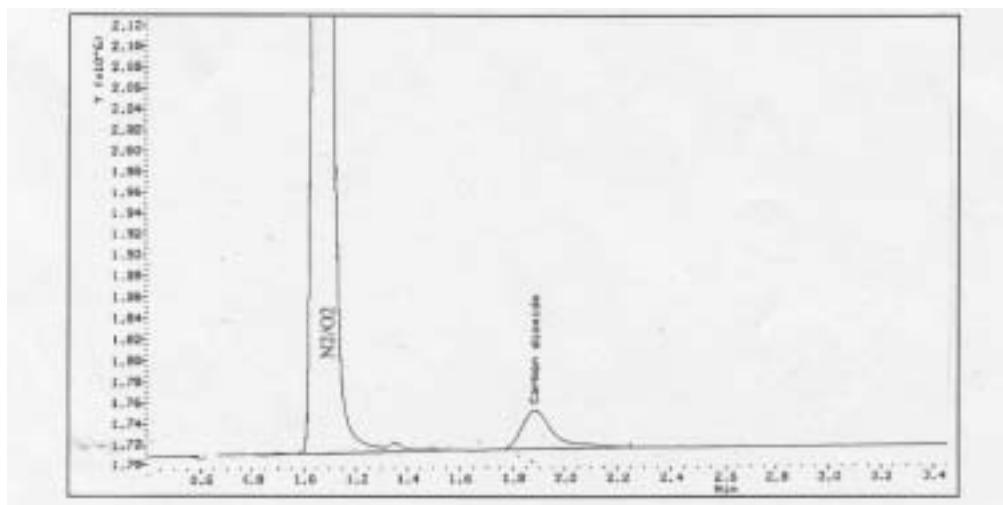


Figure 2
Carbon Dioxide Determination in Octafluorocyclobutane

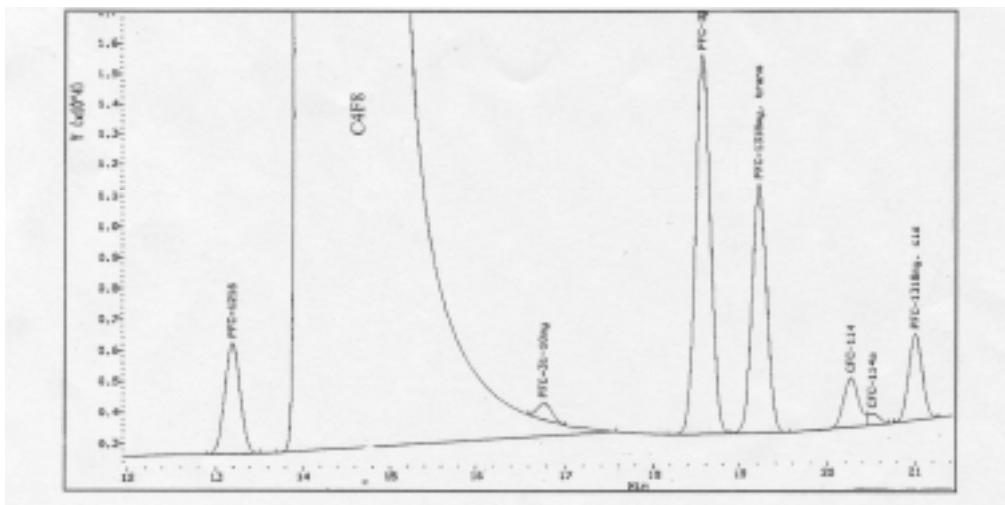


Figure 3
Determination of Organic Impurities in Octafluorocyclobutane

6.3.4.2 Obtain a continuous flow sample of the Octafluorocyclobutane source. The sample system by which the octafluorocyclobutane is passed to the instrument should consist of electroplated stainless steel tubing, zero dead-volume fittings, and be heated and purged with dry gas while not in use.

6.3.4.3 Allow the sample gas to flow through the sampling system and instrument until a stable reading is obtained.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standard 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 C3.22-1000 (Withdrawn 1104)

STANDARD FOR OXYGEN (O₂), 99.5% QUALITY

This standard 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. Originally published in 1983; previously published in 1993.

NOTICE: This document was balloted and approved for withdrawal in 2004.

1 Description

1.1 Oxygen is a colorless, odorless and oxidizing gas. It supports combustion.

2 Specifications

ASSAY: 99.5%

Impurities	Maximum Acceptable Level (ppm)*
Carbon monoxide and Carbon dioxide (CO + CO ₂)	5
Nitrogen (N ₂)	100
Nitrous oxide (N ₂ O)	2
Particles	**
Total Hydrocarbons expressed as Methane (THC)	25
Water (H ₂ O) (v/v)	1
TOTAL IMPURITIES INCLUDING RARE GASES	5,000

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

** To be determined between supplier and user.

3 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	31.999	31.999
Boiling point at 1 atm	-183°C	-297.4°F
Density of gas at 25°C (77°F) and 1 atm	1.309 kg/m ³	0.082 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	1.1049	1.1049
Density of liquid at boiling point	1142 kg/m ³	71.27 lb/ft ³

4 Analytical Procedures

4.1 *Carbon Monoxide, Carbon Dioxide, and Nitrous Oxide* — This procedure is for the determination of

carbon monoxide, carbon dioxide and nitrous oxide in oxygen using infrared spectrophotometry.

4.1.1 *Detection Limits* — 0.5 ppm (mole/mole) carbon monoxide, 0.1 ppm (mole/mole) carbon dioxide, and 0.2 ppm (mole/mole) nitrous oxide.

4.1.2 *Instrument Parameters*

4.1.2.1 10 meter variable path infrared gas cell.

4.1.2.2 Grating infrared spectrophotometer.

4.1.2.3 Bourdon Vacuum Gauge.

4.1.3 *Calibration Standards* — 10 ppm (mole/mole) carbon monoxide, 10 ppm (mole/mole) carbon dioxide and 10 ppm (mole/mole) nitrous oxide, balance oxygen.

4.1.4 *Operating Procedure*

4.1.4.1 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.

4.1.4.2 Evacuate the cell and pressurize to 50 psia with the oxygen sample. Scan the appropriate wave numbers as in 4.1.4.1.

4.1.4.3 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 2 of this Standard.

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

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

4.2.1 *Detection Limit* — 10 ppm (mole/mole).

4.2.2 *Instrument Parameters*

4.2.2.1 Column: 5A molecular sieve, 4.6 m (15 ft) by 3.2 mm (1/8 in) ss or equivalent.

4.2.2.2 Carrier Flow: 30 mL/min helium.

4.2.2.3 Sample Volume: 2.0 mL.

4.2.2.4 Temperatures:

Detector	200°C
Column Oven	21°C

4.2.3 *Calibration Standard* — 100 ppm (mole/mole) nitrogen in oxygen.

4.2.4 *Operating Procedure*

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

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

4.2.4.3 Repeat 4.2.4.1.

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

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

4.3 *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 1, 2, 3.)

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

4.3.2 *Flow Requirements*

4.3.2.1 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.

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

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

4.3.3 *Calibration Standards*

4.3.3.1 Zero oxygen with known quantity of hydrocarbons at 0.5 ppm level.

4.3.3.2 The upper level span gas not exceeding 4 times the concentration of the specification.

4.3.4 *Operating Procedure*

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

4.3.4.2 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.

4.3.4.3 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.

4.3.4.4 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 2 of this Standard.

4.4 *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 4, 5, 6.)

4.4.1 *Detection Limit* — 0.6 ppm (vol/vol) at -79°C (-100°F).

4.4.2 *Flow Requirements*

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

4.4.2.2 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.

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

4.4.4 *Operating Procedure*

4.4.4.1 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.

4.4.4.2 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.

4.4.4.3 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.

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

4.4.4.5 Correct the dewpoint reading from the measured pressure to 1 atm of pressure. The result may not exceed the specification in Section 2 of this Standard.

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

4.5.1 *Method Capability* — 99.9% Oxygen.

4.5.2 *Instrument Parameters* — See Diagram A.

4.5.2.1 *Equipment*:

1. 1-100 mL calibrated certified buret, calibrated every 0.1 mL for the top 5 mL equipped with a 3-way stopcock on the top.
2. 3-250 mL aspirator bottles.
3. 1 one-hole rubber stop.
4. 1-1/16 in OD soft copper wire made into hollow coils 3/8 in OD by 3/4 in long.
5. Sufficient 3/8 in OD tygon tubing.
6. Sufficient surgical rubber tubing.
7. 1 gallon distilled water.
8. 2 lbs technical grade ammonium chloride.
9. 1 gallon 27% ammonium hydroxide.

4.5.2.2 *Test Solution Preparation*

4.5.2.2.1 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.

4.5.3 *Equipment Assembly* — The equipment is to be assembled, as per Diagram A, in a suitable wood or metal frame so arranged that the aspirator bottle connected to the buret 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.

4.5.4 *Operating Procedure*

4.5.4.1 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 7.)

4.5.4.2 Attach rubber tubing from the regulator of the cylinder being tested to the stopcock of the buret. Rotate the stopcock so as to draw the sample into the gas measuring buret. Collect a little more than 100 mL (i.e., below the zero mark) in the buret, and then rotate the stopcock so as to shut off the oxygen flow and remove the rubber tubing attached to the intake of the buret. If the gas sample in the buret is below the zero mark raise the leveling bottle #1 so that its “water” level is even atmosphere so as to raise the “water” level in the buret to exactly zero (Diagram B, Fig. #1).

4.5.4.3 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 (Diagram B, Fig. #2). 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 buret (Diagram B, Fig. #3). 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 buret while, at the same time, gently rocking and tapping the center bottle (Diagram B, Fig. #4). This will move any bubbles which might cling to the copper coil into the gas measuring buret.

4.5.4.4 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 buret. When the levels of the leveling bottle and the gas buret are the same, read the gas purity markings on the buret at the liquid level at this point.

4.5.5 *Maintenance*

4.5.5.1 Copper coils should be added to the test bottle as required to keep the bottles completely full. The gas buret 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 buret has any liquid drops hanging in it, as this will give an erroneous purity reading.

4.5.5.2 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.



4.5.6 Standardization

4.5.6.1 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.

4.5.6.2 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.

4.6 Notes

Note 1: 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 2: 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.

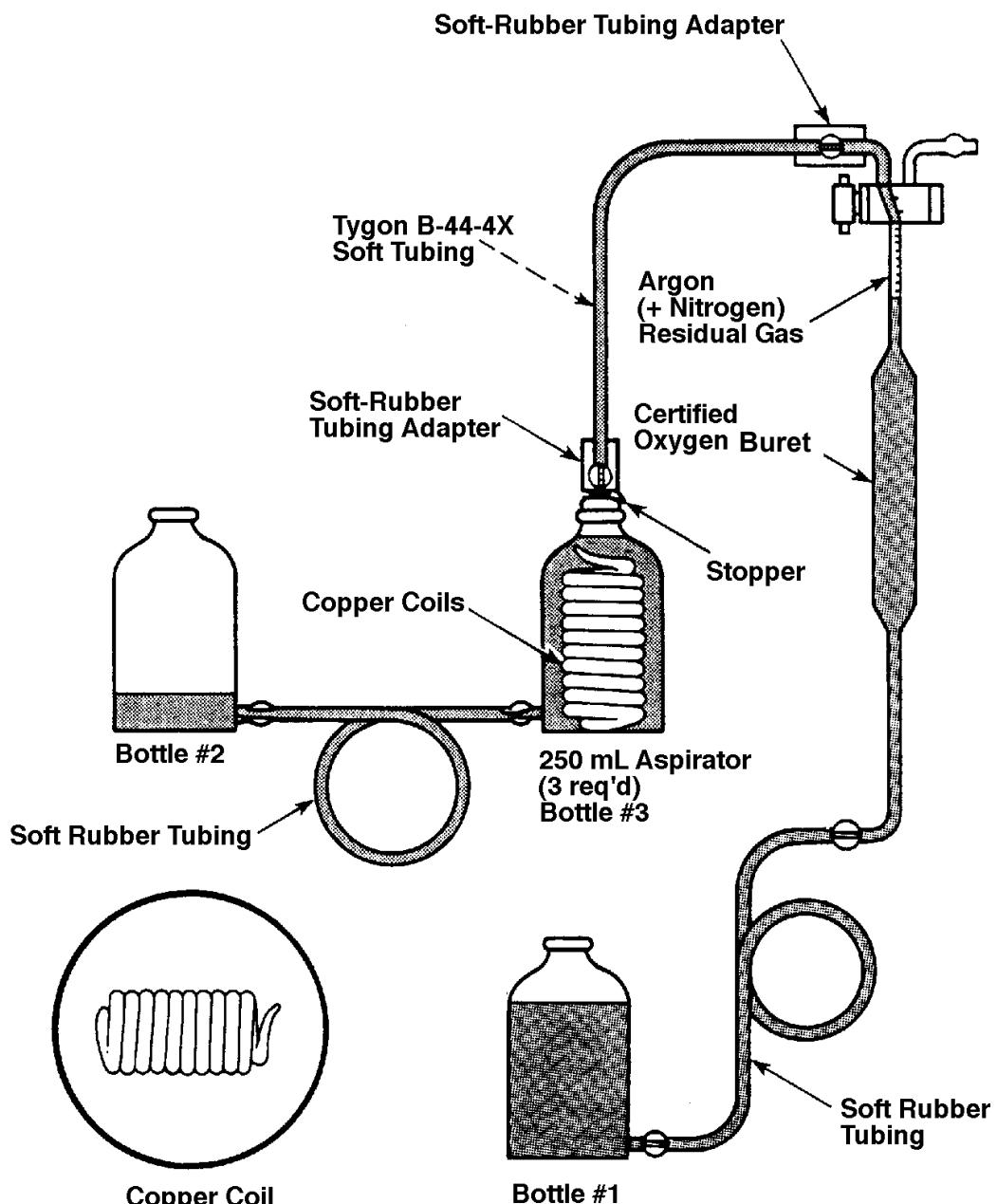
Note 3: The effective response of a flame ionization detector-equipped total hydrocarbon analyzer to different hydrocarbons can vary and must be approximated. However, the response of the most common hydrocarbon impurities in oxygen can be accurately totaled and compared to methane.

Note 4: 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 diaphragm of stainless steel or other suitable material) to accommodate the pressure restrictions of the analytical hygrometer.

Note 5: The National Institute of Standards and Technology (NIST) provides calibration services for the thermometers used in dewpoint/frostpoint hygrometers.

Note 6: This method is not applicable if other constituents in the gas will condense before water vapor, e.g., carbon dioxide and/or oil contamination.

Note 7: This procedure will be required only immediately after changing the test solution or contaminating the apparatus with air.



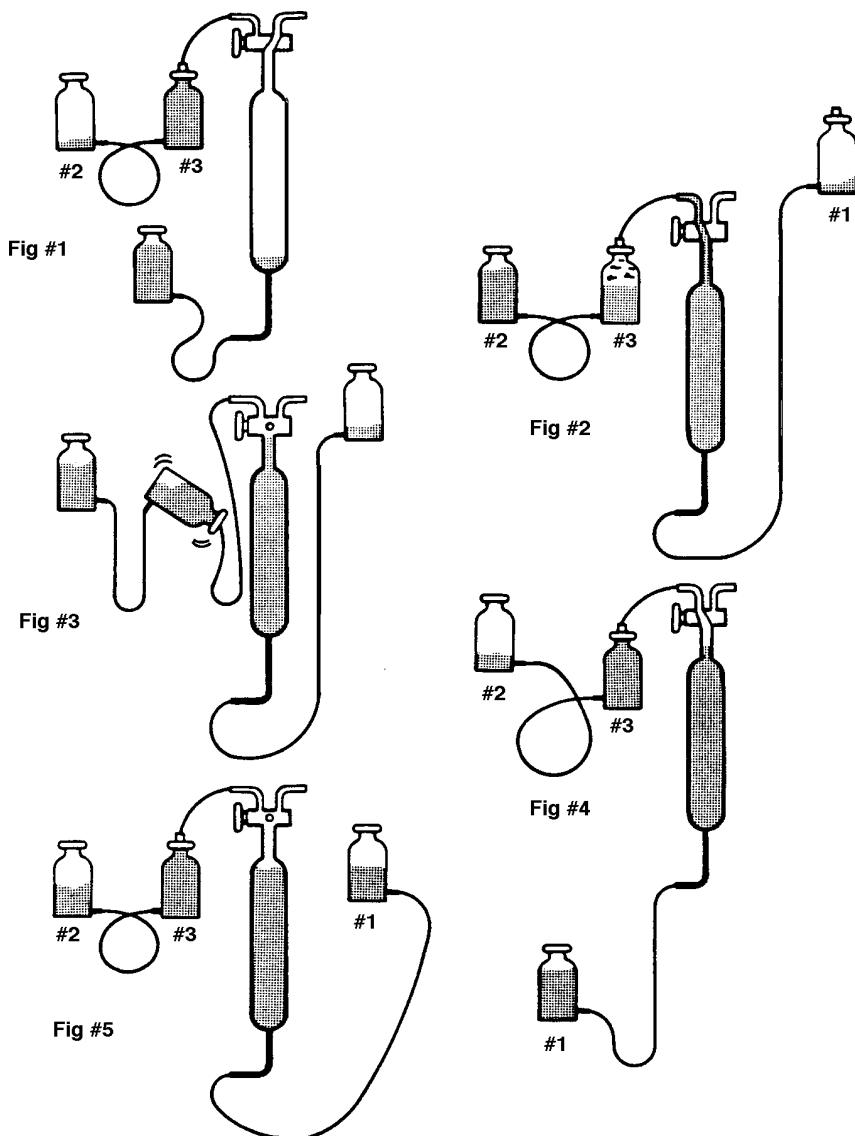


Diagram B

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

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SEMI C3.23-1000 (Withdrawn 1104)

STANDARD FOR OXYGEN (O₂), 99.98% QUALITY

This standard 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. Originally published in 1984; previously published in 1995.

NOTICE: This document was balloted and approved for withdrawal in 2004.

1 Description

1.1 Oxygen is a colorless, odorless, and oxidizing gas. It supports combustion.

2 Specifications

QUALITY: 99.98%

Impurities	Maximum Acceptable Level (ppm)*
Argon (Ar)	100
Carbon Monoxide (CO)	1
Carbon Dioxide (CO ₂)	1
Krypton (Kr)	10
Nitrogen (N ₂)	30
Nitrous Oxide (NO)	1
Particles	**
Total Hydrocarbons expressed as Methane (THC)	1
Water (H ₂ O) (v/v)	1
TOTAL IMPURITIES	146

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

** To be determined between supplier and user.

3 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	31.999	31.999
Boiling point at 1 atm	-183°C	-297.4°F
Density of gas at 25°C (77°F) and 1 atm	1.309 kg/m ³	0.082 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	1.1049	1.1049
Density of liquid at boiling point	1142 kg/m ³	71.27 lb/ft ³

4 Analytical Procedures (See Notes 1, 2)

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

4.1.1 *Detection Limit* — 25 ppm (mole/mole)

4.1.2 Instrument Parameters

4.1.2.1 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.

4.1.2.2 Carrier Flow: 45 mL/min helium.

4.1.2.3 *Sample Volume*: 1–3 mL.

4.1.2.4 Temperatures:

Detector	40°C
Column	-50°C

4.1.3 *Calibration Standard* — 50–150 ppm (mole/mole) argon, balance helium.

4.1.4 Operating Procedure

4.1.4.1 With the valve in Position A, load the sample loop. 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.

4.1.4.2 Inject oxygen sample to be tested in the same manner as in 4.1.4.1. Record the retention times and peak areas.

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Calculate the concentration of argon in the sample, using the formula below. The result may not exceed the specification Section 2 of this Standard.

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

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

4.2.1 *Detection Limit* — 0.2 ppm (mole/mole)

4.2.2 Instrument Parameters

4.2.2.1 Detector: “Luft” type or equivalent

4.2.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

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

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

4.2.2.5 Wavelength: 4.75 micrometers (4750 nm)

4.2.2.6 Wavenumber: 2100 cm⁻¹

4.2.3 *Calibration Standard* — 5 ppm (mole/mole) carbon monoxide, balance oxygen.

4.2.4 *Operating Procedure* (See Notes 5, 6.)

4.2.4.1 Open the zero gas (prepurified 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.

4.2.4.2 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.

4.2.4.3 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 2 of this Standard.

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

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

4.3.1 *Detection Limit* — 0.05 ppm (mole/mole)

4.3.2 *Instrument Parameters*

4.3.2.1 Detector: "Luft" type or equivalent

4.3.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

4.3.2.3 *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.

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

4.3.2.5 Wavelength: 4.4 micrometers (4400 nm)

4.3.2.6 Wavenumber: 2250 cm⁻¹

4.3.3 *Calibration Standard* — 5 ppm (mole/mole) carbon dioxide, balance oxygen.

4.3.4 *Operating Procedure* — (See Notes 5 and 6)

4.3.4.1 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.

4.3.4.2 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.

4.3.4.3 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 2 of this standard.

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

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

4.4.1 Detection Limits — 3 ppm (mole/mole) krypton, 3 ppm (mole/mole) nitrogen

4.4.2 Instrument Parameter

4.4.2.1 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.

4.4.2.2 Carrier Flow: 45 mL/min helium.

4.4.2.3 Sample Volume: 25 mL.

4.4.2.4 Temperatures:

Detector	40°C
Column	25°C

4.4.3 Calibration Standards — 5–15 ppm (mole/mole) krypton, 15–5 ppm (mole/mole) nitrogen, balance helium.

4.4.4 Operating Procedure

4.4.4.1 With the valve in Position A, load the sample loop. 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.

4.4.4.2 Inject the sample to be tested in the same manner as 4.4.4.1. Record the retention times and peak areas.

4.4.4.3 Repeat 4.4.4.1.

4.4.4.4 Calculate the concentrations of krypton and nitrogen using the formula below. The result may not exceed the specification in Section 2 of this standard.

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

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

4.5.1 Detection Limit — 0.05 ppm (mole/mole)

4.5.2 Instrument Parameters

4.5.2.1 Detector: “Luft” type or equivalent

4.5.2.2 10" Infrared Gas Cell or gas cell with equivalent sensitivity

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

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

4.5.2.5 Wavelength: 4.5 micrometers (4500 nm)

4.5.2.6 Wavenumber: 2222 cm⁻¹

4.5.3 Calibration Standard: 5 ppm (mole/mole) nitrous oxide, balance oxygen.

4.5.4 Operating Procedure — (See Notes 5 and 6)

4.5.4.1 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 VI. After a constant readout is observed, adjust the zero control knob of the analyzer to set the absorbance output to read zero.

4.5.4.2 Open the calibration gas standard cylinder valve. Close VI 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.

4.5.4.3 Introduce the oxygen sample into the analyzer by closing valves VI 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 2 of this standard.

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

4.6 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 7, 8.)

4.6.1 Detection Limit — 0.1 ppm (mole/mole)

4.6.2 Flow Requirements

4.6.2.1 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.

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

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

4.6.3 Calibration Standards

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

4.6.3.2 Upper level balance oxygen span gas of not more than five times the concentration specified in Section 2 of this Standard.

4.6.4 Operating Procedure

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

4.6.4.2 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.

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

4.6.4.4 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.

4.7 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 Note 9.)

4.7.1 Detection Limit — 0.6 ppm (vol/vol) at -79°C (-100°F)

4.7.2 Flow Requirements

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

4.7.3 Calibration Standard — A calibration thermometer designed to indicate temperatures in the -79°C (100°F) range is required.

4.7.4 Operating Procedure

4.7.4.1 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.

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

4.7.4.3 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.

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

4.7.4.5 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 2 of this standard.

4.8 Notes

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 not contain more than 10% of the specified value of the component of interest, unless otherwise stated.

Note 3: A flow restrictor should be installed on the vent from port 8 to match the pressure drop of the detector.

Note 4: A flow controller is required on each carrier inlet.

Note 5: An example of an operating procedure is outlined below for the sample system shown in Figure 1. This procedure is appropriate only when the infrared gas cell is designed to withstand the 200 psig sample pressure.

Note 6: Operating procedures for other non-dispersive infrared analyzers vary depending on manufacturer. Refer to individual instrument vendor instructions in each case.

Note 7: 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 8: The effective response of a flame ionization detector-equipped total hydrocarbon analyzer can vary among different hydrocarbons and must be approximated. However, the response of the most common hydrocarbon impurities can be accurately totaled and compared to methane.

Note 9: The sampling system and hygrometer must be designed to operate at the sample pressure, or the sample pressure must be reduced by a regulator with a diaphragm of stainless steel or other suitable material.

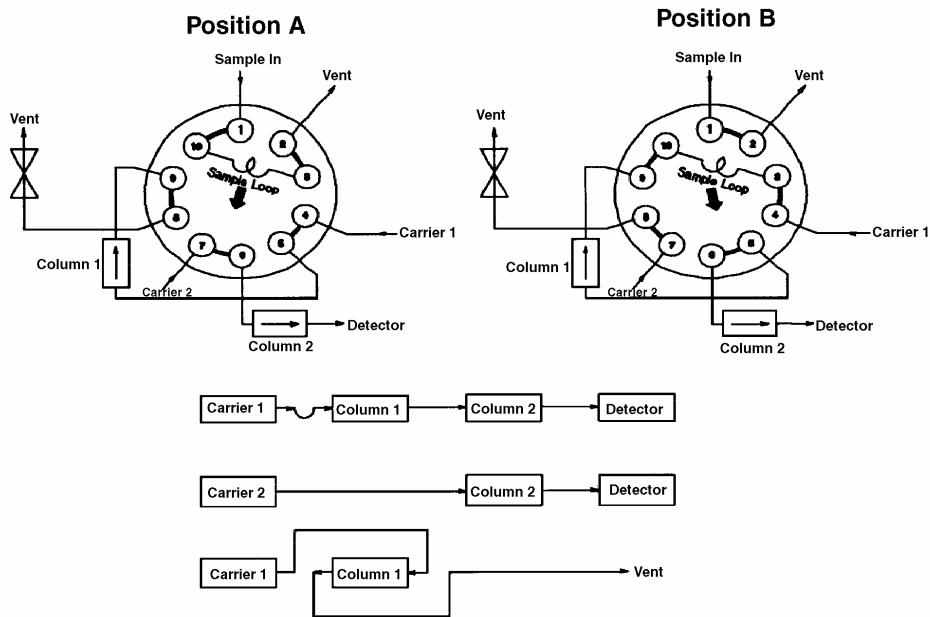


Figure 1

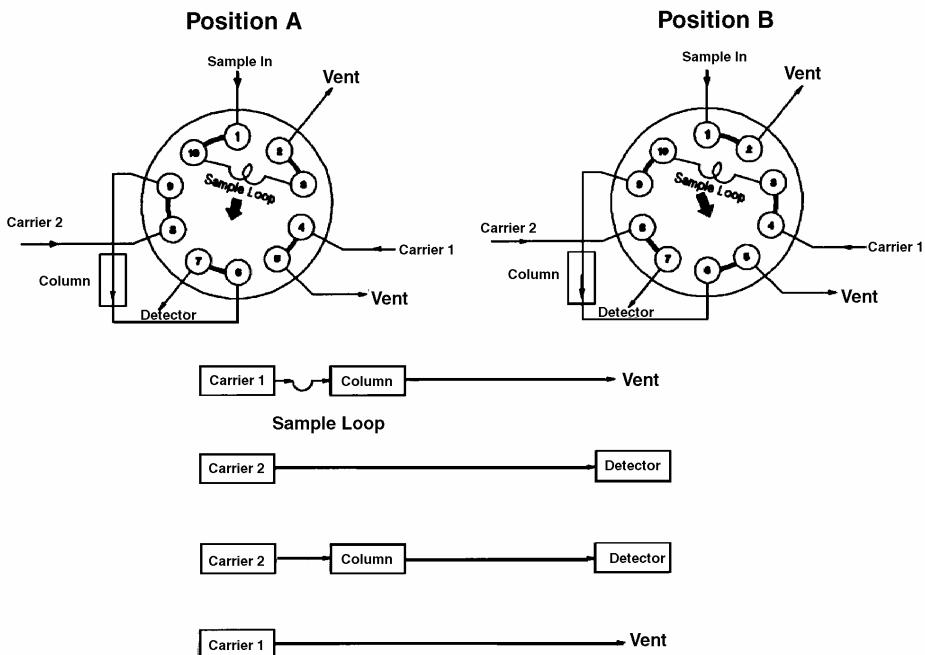
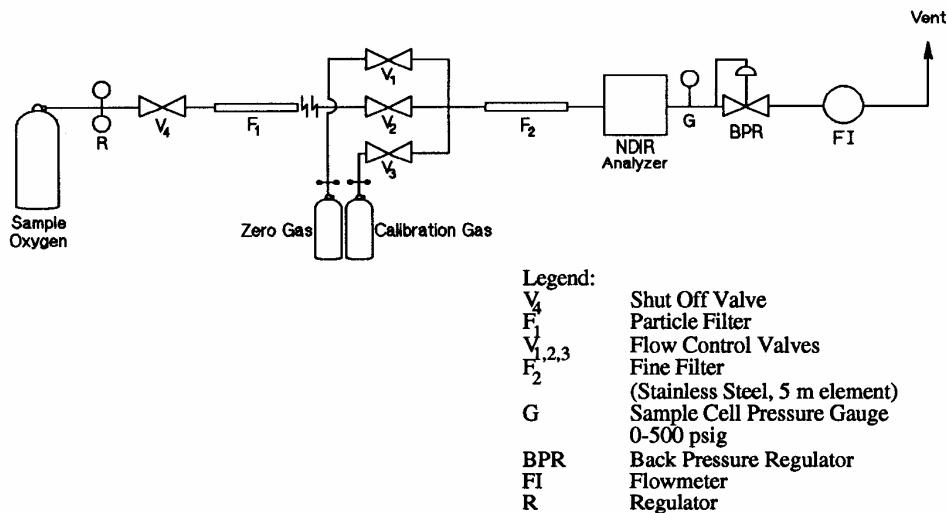


Figure 2



Note: All Components in contact with the sample must be constructed of stainless steel, teflon, or glass.

Figure 3
Sample System for NDIR Analyzer

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

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SEMI C3.41-0703 (Withdrawn 1104)

I STANDARD FOR OXYGEN (O₂), BULK, 99.9998% QUALITY

This standard 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. Originally published in 1990; previously published July 2003.

NOTICE: This document was balloted and approved for withdrawal in 2004.

Density of liquid at boiling point	1142 kg/m ³	71.27 lb/ft ³
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1 Description

1.1 Oxygen is a colorless, odorless, and oxidizing gas. It supports combustion.

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.

2 Specifications

QUALITY: 99.9998%

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

Impurities	Maximum Acceptable Level (ppm) (See Note 1.)
Argon (Ar)	1.0
Carbon Dioxide (CO ₂)	0.1
Carbon Monoxide (CO)	0.1
Hydrogen (H ₂)	0.1
Nitrogen (N ₂)	0.5
Particles	(See Note 2.)
Total Hydrocarbons (as CH ₄)	0.1
Water (H ₂ O) (v/v)	0.1
TOTAL SPECIFIED IMPURITIES	2.0

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

NOTE 2: To be determined between user and supplier.

3 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	31.999	31.999
Boiling point at 1 atm	-183.0°C	-297.4°F
Density of gas at 25°C (77°F) and 1 atm	1.309 kg/m ³	0.082 lb/ft ³
Specific gravity of gas	1.1049	1.1049

4 Analytical Procedures

4.1 *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 1).

4.1.1 *Detection Limit* — 0.1 ppm

4.1.2 *Instrument Parameters*

4.1.2.1 *Column(s)*:

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

3 m (10 ft) by 3.2 mm (1/8 in.) Oxy-trap (See Figure 1.)

4.1.2.2 *Carrier Flow*: 44 cc/min

4.1.3 *Sample Volume*: 0.1 mL

4.1.3.1 *Temperatures*:

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

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

4.1.5 *Operating Procedures*

4.1.5.1 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.

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

4.1.5.3 Repeat 4.1.5.1.

4.1.5.4 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 2 of this standard.

Figure 2 Oxygen trap conditioned per manufacturer's recommendations using hydrogen, carbon monoxide, or blend in helium.

4.2 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.

4.2.1 Detection Limit — 0.02 ppm

4.2.2 Instrument Parameters

4.2.3 Column(s): 2.3 m (91 in.) by 3.2 mm (1/8 in.) HayeSep Db

4.2.3.1 Carrier Flow: 50 cc/min nitrogen

4.2.3.2 Sample Volume: 5 mL

4.2.3.3 Temperatures:

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

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

4.2.5 Operating Procedures

4.2.5.1 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.

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

4.2.5.3 Repeat Section 4.2.5.1

4.2.5.4 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 2 of this standard.

Figure 3 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.

4.3 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.

4.3.1 Detection Limit — 0.002 ppm

4.3.2 Instrument Parameters

4.3.2.1 Column(s): 2 m (80 in.) by 3.2 mm (1/8 in.) Molecular Sieve 13×

4.3.2.2 Carrier Flow: 20 cc/min air

4.3.2.3 Sample Volume: 1 mL

4.3.2.4 Temperatures:

Detector	265°C
Column	105°C

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

4.3.4 Operating Procedures

4.3.4.1 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.

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

4.3.4.3 Repeat Section 4.3.4.1

4.3.4.4 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 2 of this standard.

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

4.4.1 Detection Limit — 0.05 ppm

4.4.2 Instrument Parameters

4.4.2.1 Flow Requirements:

Carrier Flow Rate: per manufacturer's recommendations

Purity: less than 0.05 ppm total hydrocarbons

Sample Flow Rate: manufacturer's recommendations

4.4.3 Calibration Standards — 0.1–1 ppm methane in oxygen.

4.4.4 Operating Procedures

4.4.4.1 Flow settings should not be changed once established.

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

4.4.4.3 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 2 of this standard.

4.5 *Water* — This procedure is for the determination of trace moisture (water) in O₂ using a continuous flowing piezoelectric hygrometer (see Note 4).

4.5.1 *Detection Limit* — 5 ppb

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

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

4.5.4 *Operating Procedure*

4.5.4.1 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.

4.5.4.2 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.

4.5.4.3 Determine the moisture content of the oxygen sample by comparing the reading to calibration curve. The result may not exceed the specification in Section 2 of this standard.

NOTE 4: 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.

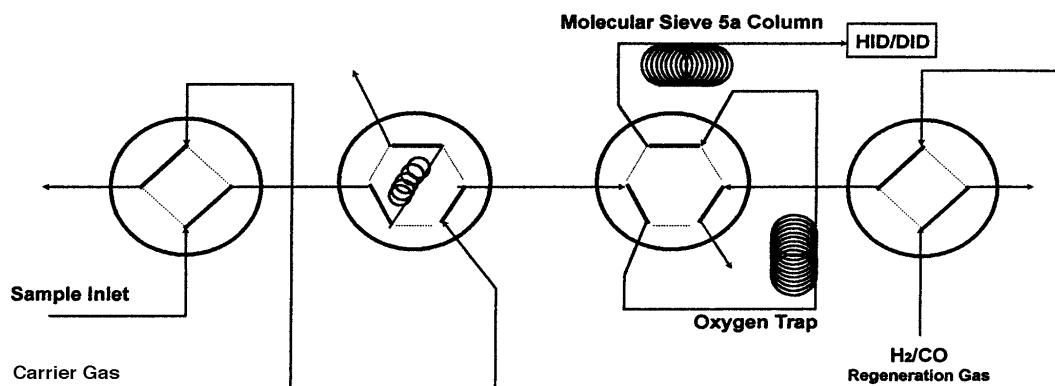


Figure 4
Analysis of Nitrogen and Argon in Oxygen Using Oxygen Trap

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 C3.6-0701

SPECIFICATION FOR PHOSPHINE (PH₃) IN CYLINDERS, 99.98% QUALITY

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on April 30, 2001. Initially available at www.semi.org May 2001; to be published July 2001. Originally published in 1981; previously published in 1995.

1 Description

1.1 Phosphine is a highly toxic, flammable, colorless gas with an odor of decaying fish. In high concentrations, it is pyrophoric. It is shipped as a liquid in cylinders under its own vapor pressure.

2 Specifications

QUALITY: 99.98%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Arsine (AsH ₃)	10
Carbon dioxide (CO ₂)	10
Carbon Monoxide (CO)	1
Heavy Metals	**
Hydrogen (H ₂)	100
Nitrogen (N ₂)	50
Oxygen (O ₂) + Argon (Ar)	5
Particles	**
Hydrocarbons expressed as Methane (C ₁ -C ₂)	4
Water (H ₂ O) (v/v)	1
TOTAL LISTED IMPURITIES	181

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

** To be determined between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	33.997	33.997
Boiling point at 1 atm	-87.7°C	-125.9°F
Density of gas at 25°C (77°F) and 1 atm	1.402 kg/m ³	0.0875 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	1.146	1.146
Density of liquid at boiling point	744 kg/m ³	46.45 lb/ft ³

4 Analytical Procedures

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

4.1.1 *Detection Limit* — 0.1 ppm each impurity.

4.1.2 *Instrument Parameters*

4.1.2.1 *Column* — Porapak QS, 3.5 m (12 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID ss or equivalent.

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

4.1.2.3 *Support Gases*

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

Air: 500 mL/min.

4.1.2.4 *Temperatures:*

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

4.1.2.5 *Sample Volume* — 3 mL.

4.1.3 *Calibration Standards* — 1–10 ppm each: carbon monoxide, carbon dioxide, methane, ethane*, ethylene*, and acetylene*, balance helium. *These components are for reference only.

4.1.4 *Operating Procedure*

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

4.1.4.2 Inject the sample to be tested in the same manner as the calibration standard. Vent the phosphine after the ethylene peak. Record the retention times and peak areas.

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Calculation of Concentrations

4.1.4.4.1 Carbon Monoxide and Carbon Dioxide — Compare the average peak areas for the calibration standard to those of the phosphine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed the specification in Section 2 of this standard.

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

4.1.4.4.2 Total Hydrocarbons Expressed as Methane (C_1-C_2) — Compare the peak area of methane in the calibration standard with the sum of the areas of the hydrocarbons detected in the phosphine sample. Calculate the concentration of total hydrocarbons using the formula below. The results may not exceed the specifications in Section 2 of this standard.

$$\frac{\text{Sum of areas for all Hydrocarbons detected}}{\text{Standard Peak Area for Methane}} \times \frac{\text{Concentration of Methane in Standard}}{\text{Concentration of Sample}} = \frac{\text{THC Concentration of Sample}}{\text{Concentration of Standard}}$$

4.2 Hydrogen, Nitrogen, and Oxygen + Argon, Arsine — This procedure is for the determination of hydrogen, nitrogen, oxygen + argon, and arsine in phosphine using a gas chromatograph with a discharge ionization detector. (See Note 2 and Figure 2.)

4.2.1 Detection Limits — 100 ppb (mol/mol) hydrogen, 100 ppb (mol/mol) oxygen + argon, 500 ppb (mol/mol) nitrogen, 5 ppm (mol/mol) arsine.

4.2.2 Instrument Parameters

4.2.2.1 Columns:

Column 1:	Porapak QS, 2.5 m (8 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID ss or equivalent
Column 2:	Molecular Sieve 5A, 3 m (10 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID ss or equivalent

4.2.2.2 Carrier Flow — 25 mL/min helium.

4.2.2.3 Temperatures

Detector	10°C
Injector	30°C
Oven	40°C

4.2.2.4 Sample Volume — 1 mL.

4.2.3 Calibration Standard — 5–50 ppm (mol/mol) nitrogen, 1–10 ppm (mol/mol) oxygen, 1–10 ppm (mol/mol) argon, 10–100 ppm (mol/mol) hydrogen, 5–10 ppm (mol/mol) arsine, balance helium.

4.2.4 Operating Procedure

4.2.4.1 Determination of the Switching Time — Inject the calibration sample in 4.2.3 above and note the time when the hydrogen peak appears through detector B. Switching valve 1 at that time will give an arsine peak through detector A and hydrogen, oxygen + argon, and nitrogen peaks through detector B.

4.2.4.2 Inject the calibration standard into the column using the same gas valve. Switch at the time determined in 4.2.4.1 and record retention times and peak areas. Order of elution is hydrogen, oxygen + argon, and nitrogen on detector B. Order of elution is phosphine and arsine on detector A.

4.2.4.3 Inject the sample being tested in same manner as the calibration standard. Record the retention times and peak areas.

4.2.4.4 Repeat 4.2.4.2.

4.2.4.5 Compare the average peak areas of the calibration standard to those of the phosphine sample being tested. Calculate the concentration of each impurity, using the formula below. The results may not exceed the specifications in Section 2 of this standard.

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

4.3 Water — This procedure is for the determination of moisture in phosphine using continuous flow electrolysis of water in a phosphorous pentoxide (P_2O_5) cell, modified as per the manufacturer's recommendation, for use with phosphine.

4.3.1 Detection Limit — 0.5 ppm (vol/vol).

4.3.2 Instrument Parameters

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

4.3.3 Calibration Standards — Construct a calibration curve which contains at least three points covering the range of interest. Verify the standards employed independently on a condensation dew point/frost point hygrometer. Standards are prepared in nitrogen.

4.3.4 Operating Procedure

4.3.4.1 Obtain representative sample of gas to analyze and direct to unit as with the standards.

4.3.4.2 Determine the moisture content in sample gas by comparing the indicated concentration with the calibration curve constructed in 4.3.4.1. The result may not exceed the specification in Section 2 of this standard.

4.4 Notes

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

NOTE 2: Carrier gas purity of helium must be 99.9999%.

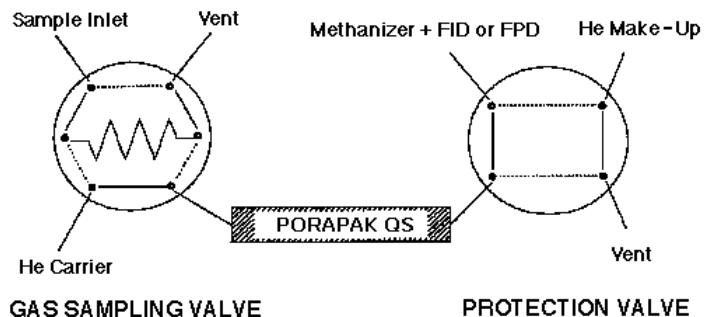


Figure 1
G.C. Configuration

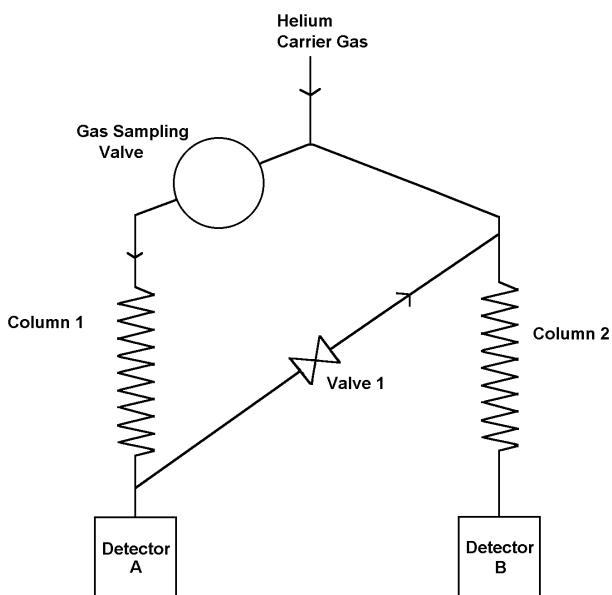


Figure 2
Column Configuration for H_2 , N_2 , and $\text{O}_2 + \text{Ar}$, AsH_3

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

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SEMI C3.54-0200

GAS PURITY GUIDELINE FOR SILANE (SiH_4)

This guideline 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 15, 1999. Initially available at www.semi.org January 2000, to be published February 2000. Originally published in 1997.

1 Description

1.1 Silane is a pyrophoric, flammable colorless gas.

2 Specifications

Quality: 99.994%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)</i>
Arsine (AsH_3)	1 ppb
Carbon Monoxide (CO)	0.1
Carbon Dioxide (CO_2)	0.1
Chlorosilanes (ionizable chlorides) including HC1, reported as chloride (C1)	1
Non-methane Hydrocarbons ($\text{C}_2 - \text{C}_4$)	0.1 total
Hydrogen (H_2)	50
Methane (CH_4)	0.1
Nitrogen (N_2)	1
Phosphine (PH_3)	0.1 ppb
Disiloxane ($\text{H}_3\text{SiOSiH}_3$)	1
Methyl Silane ($\text{SiH}_3\text{-CH}_3$)	1
Disilane (Si_2H_6)	1
Aluminum (Al)	0.2 ppba (See NOTE 1.)
Antimony (Sb)	0.2 ppba (See NOTE 1.)
Arsenic (As)	0.2 ppba (See NOTE 1.)
Boron (B)	0.02 ppba (See NOTE 1.)
Gallium (Ga)	0.2 ppba (See NOTE 1.)
Phosphorus (P)	0.02 ppba (See NOTE 1.)
Calcium (Ca) + Chromium (Cr) + Copper (Cu) + Iron (Fe) + Potassium (K) + Lithium (Li) + Magnesium (Mg) + Manganese (Mn) + Molybdenum (Mo) + Sodium (Na) + Nickel (Ni) + Lead (Pb) + Zinc (Zn)	1.0 ppba total (See NOTE 1.)
Water (H_2O) (vol/vol)	1
Particles	(See NOTE 2.)
TOTAL SPECIFIED IMPURITIES	56.4033

NOTE 1: To be determined between supplier and user.

NOTE 2: ppba is defined to be atoms of impurity per 10^9 atoms of silicon.

3 Electrical Specification

Resistivity	greater than 10,000 ohm-cm (n-type)
-------------	-------------------------------------

4 Physical Constants

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	32.112	32.112
Boiling point at 1 atm.	-112°C	-169.6°F
Density of gas at 21.1°C (70°F) and 1 atm.	1.342 kg/m³	0.0839 lb/ft³
Specific gravity of gas at 21.1°C and 1 atm. (air = 1)	1.114	1.114
Density of liquid at boiling point.	711 kg/m³	44.39 lb/ft³

5 Procedures (See NOTE 1)

NOTE 1: Standardized test methods are being developed for all parameters at the purity level indicated. Until standardized test methods are published, test methodology shall be determined by user and producer.

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SEMI C3.55-0200

STANDARD FOR SILANE (SiH₄), BULK

This standard 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 15, 1999. Initially available at www.semi.org January 2000; to be published February 2000.

1 Description

1.1 Silane is a pyrophoric, flammable colorless gas.

2 Specifications

QUALITY: 99.994%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)</i>
<i>Gas Phase</i>	
Carbon Monoxide (CO)	0.1
Carbon Dioxide (CO ₂)	0.1
Chlorosilanes (ionizable chlorides) including HCl, reported as chloride (Cl ⁻)	1
Non-methane Hydrocarbons (C ₂ -C ₄)	0.1 total
Hydrogen (H ₂)	50
Methane (CH ₄)	0.1
Nitrogen (N ₂)	1
Argon (Ar)	1
Disiloxane (H ₃ SiOSiH ₃)	1
Methyl Silane (SiH ₃ -CH ₃)	1
Disilane (Si ₂ H ₆)	1
Water (H ₂ O)(vol/vol)	1
Particles	(See NOTE 1.)
Total Specified Impurities	57.4
<i>Deposited Layer</i>	
Carbon (C)	0.5 ppma (See NOTE 2.)
Oxygen (O)	1 ppma (See NOTE 2.)
Aluminum (Al)	0.2 ppba (See NOTE 2.)
Antimony (Sb)	0.2 ppba (See NOTE 3.)
Arsenic (As)	0.2 ppba (See NOTE 3.)
Boron (B)	0.2 ppba (See NOTE 3.)
Gallium (Ga)	0.2 ppba (See NOTE 3.)
Phosphorus (P)	0.2 ppba (See NOTE 3.)
Chromium (Cr)+ Copper (Cu)+ Iron (Fe)+ Nickel (Ni)+ Zinc (Zn)	1.0 ppba total (See NOTE 3.)

NOTE 1: To be determined between supplier and user.

NOTE 2: ppma is defined to be atoms of impurity per 10⁶ atoms of silicon.

NOTE 3: ppba is defined to be atoms of impurity per 10⁹ atoms of silicon.

3 Electrical Specification

Resistivity	greater than 2,000 ohm-cm (n-type)
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4 Physical Constants

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	32.112	32.112
Boiling point at 1 atm.	-112°C	-169°F
Density of gas at 21.1°C (70°F) and 1 atm.	1.342 kg/m ³	0.0839 lb/ft ³
Specific gravity of gas at 21.1°C and 1 atm (air = 1)	1.114	1.114
Density of liquid at boiling point	711 kg/m ³	44.39 lb/ft ³

5 Analytical Procedures (see NOTE 1)

5.1 *Carbon Monoxide, Carbon Dioxide, Argon, Nitrogen and Methane* — This procedure is for the determination of carbon monoxide, carbon dioxide, argon, nitrogen and methane in silane using a gas chromatograph with a discharge ionization detector.

5.1.1 *Detection Limit* — 30 ppb (mole/mole)

5.1.2 *Instrument Parameters*

5.1.2.1 *Column:*

HAYESEP D 60/80, 6.5 m (21.3 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel; or equivalent.

5.1.2.2 *Carrier Flow* — 25 mL/min helium

5.1.2.3 *Sample Volume* — 1 mL

5.1.2.4 *Temperatures:*

Detector	140°C
Column Oven	40°C for 4 min., to 120°C at 10°C/min., hold 120°C for 8 min.
Injection Temperature	45°C

5.1.3 *Calibration Standards* — 1 ppm (mole/mole) for all components to be tested, balance helium.

5.1.4 Operating Procedures

5.1.4.1 Inject the calibration standard and sample as described in Section 6.6 of SEMI C3. The order of elution is nitrogen, argon, carbon monoxide, methane, and carbon dioxide. (See NOTE 2.)

5.1.4.2 Compare the average peak area of the calibration standard to that of the silane sample being tested. Calculate the concentrations of the analytes as described in Section 6.8 of SEMI C3. The result shall be reported in the unit of concentration needed and to the number of significant digits of the variable with the least number of significant digits. This value may not exceed that specified in Section 2.

5.2 *Total Chlorides* — This procedure is for the determination of total chlorides in silane by titration of a hydrolyzed sample of silane. (See NOTE 1.)

5.2.1 *Detection Limit* — 0.5 ppm (mole/mole)

5.2.2 *Instrument Parameters* — See Figure 1.

5.2.2.1 Equipment

5.2.2.1.1 Safety purge regulator with proper fittings.

5.2.2.1.2 Flow meter capable of measuring 0.25 standard liters per minute (0.5 SCF/hour) (Brooks or equivalent), calibrated for silane.

5.2.2.1.3 One cylinder of nitrogen with regulator.

5.2.2.1.4 One ice bath.

5.2.2.1.5 Three magnetic stirring bars.

5.2.2.1.6 Two 2000 mL heavy-duty sidearm flasks (Fisher Cat. No. 10-181G or equivalent).

5.2.2.1.7 One #9 one-hole rubber stopper.

5.2.2.1.8 One #9 two-hole rubber stopper.

5.2.2.1.9 Two Pyrex-brand tubes with fritted cylinders (Fisher Cat. No. 11-138B or equivalent).

5.2.2.1.10 One gas washing bottle with fritted cylinder, 125 mL cap. (Fisher Cat. No. 03-040A or equivalent).

5.2.2.1.11 One buret, 50 mL capacity with Teflon stopcock (Fisher Cat. No. 03-700-22C or equivalent).

5.2.2.1.12 One buret stand (Fisher Cat. No. 14-688 or equivalent).

5.2.2.1.13 Miscellaneous clamps, support stands, and rubber hose.

5.2.2.1.14 Two 250 mL Erlenmeyer flasks.

5.2.2.2 Reagents

5.2.2.2.1 Potassium hydroxide flakes, technical (Fisher Cat. No. P-246 or equivalent).

5.2.2.2.2 Mercuric nitrate crystal (Fisher Cat. No. M-168 or equivalent).

5.2.2.2.3 Sodium chloride crystal (Fisher Cat. No. S-271 or equivalent).

5.2.2.2.4 Diphenyl carbazole (Fisher Cat. No. D-86 or equivalent).

5.2.2.2.5 Bromophenol blue (Fisher Cat. No. B-392 or equivalent).

5.2.2.2.6 Nitric acid (Fisher Cat. No. AA-200 or equivalent).

5.2.2.2.7 Ethanol, denatured (Fisher Cat. No. AA-407 or equivalent).

5.2.3 Operating Procedure

5.2.3.1 Assemble apparatus as shown in Figure 1 and purge entire system with nitrogen for 30 minutes.

5.2.3.2 Fill each 2000 mL sidearm flask with about 1700 mL of 15% potassium hydroxide solution (15 g KOH per each 100 mL water).

5.2.3.3 Fill the gas washing bottle with about 75 mL of deionized water that has been degassed.

5.2.3.4 Pass exactly one cubic foot of silane through the system at a rate of 0.5 cubic feet per hour.

5.2.3.5 Stop silane flow and purge system for 30 minutes.

5.2.3.6 Remove gas washing bottle from system and transfer contents quantitatively and with the aid of three 25 mL deionized water washings, to a 250 mL Erlenmeyer flask.

5.2.3.7 Add a few drops of indicator solution, 5 g diphenyl carbazole plus 0.5 g bromophenol blue dissolved in 750 mL ethanol, plus 250 mL deionized water.

5.2.3.8 Add, in a drop-wise fashion, sufficient 0.2 N HNO₃ (13 mL concentrated HNO₃, diluted to 1 L in deionized water) in order to just turn the solution to yellow from purple.

5.2.3.9 Titrate with a solution of mercuric nitrate Hg(NO₃)₂, (diluted to 1000 mL in deionized water; then 50 mL diluted to 500 mL in deionized water) which has been previously standardized to 5.0 mL of a sodium chloride solution (approximately 165 ng NaCl, accurately weighed, dissolved in 100 mL deionized water).

5.2.4 Calibration

5.2.4.1 Run titration blank consisting of 150 mL deionized water.

5.2.4.2 Calculate the concentration of chloride, using the formula below. The result may not exceed the specification in Section 2 of this standard.

$$\frac{\text{mL}_{\text{sample}} - \text{mL}_{\text{blank}}}{\text{mL}_{\text{standard}} - \text{mL}_{\text{blank}}} \times \frac{\text{wt. NaCl(g)}}{100.0\text{mL}} \times \frac{60.66\%\text{Cl} -}{100\%\text{NaCl}} \times$$

$$\frac{100\text{mL}}{1\text{L}} \times \frac{0.0051}{35.453\text{g/mole}} \times \frac{0.082\text{atmL/moleK}}{28.316\text{L}} \times$$

$$1 \times 10^6 \text{ ppm} = \text{ppm Cl-}$$

5.3 Hydrocarbons C₂ – C₄ — This procedure is for the determination of hydrocarbons (methane, ethane, and propane) in silane using a gas chromatograph with a flame ionization detector.

5.3.1 Detection Limit — 50 ppb (mole/mole)

5.3.2 Instrument Parameters

5.3.2.1 Columns:

Column 1:	VZ-10, 60/80, 2 m (6.6 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel;
Column 2:	VZ-10, 60/80, 1 m (3.3 ft) by 3.2 mm (1/8 in) OD by 2.2 mm (0.085 in) ID stainless steel;

or equivalent.

5.3.2.2 Carrier Flow — 30 mL/min nitrogen

5.3.2.3 Sample Volume — 10 mL

5.3.2.4 Temperatures:

Detector	150°C
Column Oven	45°C

5.3.3 Calibration Standards — 1 ppm (mole/mole) for all components to be tested, balance helium.

5.3.4 Operating Procedures — (see SEMI C3, Section 6, Gas Chromatography for standard procedures).

5.3.4.1 Inject the calibration standard and sample as described in Section 6.6 of SEMI C3. The order of elution is methane, ethane, propane and butane (see NOTE 2).

5.3.4.2 Compare the average peak area of the methane peak in the calibration standard to that of the hydrocarbons found in the silane sample being tested. Calculate the concentrations of the analytes as described in Section 6.8 of SEMI C3 based on the methane response. The result shall be reported in the unit of concentration needed and to the number of

significant digits of the variable with the least number of significant digits. This value may not exceed that specified in Section 2.

5.4 Hydrogen — This procedure is for the determination of hydrogen in silane using a gas chromatograph with a thermal conductivity detector.

5.4.1 Detection Limit — 15 ppm (mole/mole)

5.4.2 Instrument Parameters

5.4.2.1 Columns:

Molecular sieve 5A, 2.4 m (8 ft) by 6.4 mm (1/4 in) OD by 4.7 mm (0.185 in) ID, or equivalent.

5.4.2.2 Carrier Flow — 30 mL/min argon

5.4.2.3 Sample Volume — 1.0 mL

5.4.2.4 Temperatures:

Detector	40°C
Column Oven	40°C

5.4.3 Calibration Standards — 20–100 ppm (mole/mole), balance argon.

5.4.4 Operating Procedures — (see SEMI C3, Section 6, Gas Chromatography for standard procedures)

5.4.4.1 Inject the calibration standard and sample as described in Section 6.6 of SEMI C3. (See NOTE 2.)

5.4.4.2 Compare the average peak area of the hydrogen peak in the calibration standard to that of the hydrogen found in the silane sample being tested. Calculate the concentrations of the analytes as described in Section 6.8 of SEMI C3 based on the methane response. The result shall be reported in the unit of concentration needed and to the number of significant digits of the variable with the least number of significant digits. This value may not exceed that specified in Section 2.

5.5 Disiloxane, Disilane and Methyl Silane — This procedure is for the determination of disiloxane, disilane and methyl silane in silane using a gas chromatograph with flame ionization detector.

5.5.1 Detection Limit — 0.1 ppm (mole/mole)

5.5.2 Instrument Parameters

5.5.2.1 Column:

28% DC-200 on Chromosorb PAW, 45/60 mesh, 14 m (46 ft) SS, or equivalent.

5.5.2.2 Carrier Flow — 30 mL/min helium

5.5.2.3 Sample Volume — 2 mL

5.5.2.4 Temperatures — Column 60°C

5.5.2.5 *Calibration Standards* — 1–5 ppm (mole/mole)

5.5.3 *Operating Procedure* — (see SEMI C3, Section 6, Gas Chromatography for standard procedures).

5.5.3.1 Inject the calibration standard and sample as described in Section 6.6 of SEMI C3. The order of elution is silane matrix, methyl silane, siloxane (for information only) and disilane. (See NOTE 2.)

5.5.3.2 Compare the average peak area of the components being measured in the calibration standard to that of the same analytes found in the silane sample being tested. Calculate the concentrations of the analytes as described in Section 6.8 of SEMI C3. The result shall be reported in the unit of concentration needed and to the number of significant digits of the variable with the least number of significant digits. This value may not exceed that specified in Section 2.

5.6 *Water* — This procedure is for the determination of trace moisture (water) in silane using a vibrating quartz hygrometer.

5.6.1 *Detection Limit* — 0.1 ppm (vol/vol)

5.6.2 *Instrument Parameters*

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

5.6.3 *Calibration Standards* — Check the hygrometer calibration with silane using an external permeation device. Construct a calibration curve which contains at least three points covering the range of interest. The standards employed will have been verified independently on a condensation dewpoint/frostpoint hygrometer. (See Figure 2.)

5.6.4 *Operating Procedure* — At the first start-up of the hygrometer, calibrate it with moisture standards in silane. Then check the calibration once a month and modify the hygrometer if necessary. Direct standard to unit with stainless lines which have been purged. Note that silane is spontaneously flammable. Thus the experimental set-up should be leak-free and thoroughly purged before the introduction of silane. Obtain representative sample of the gas to be analyzed and direct to the unit. Wait until the equilibrium and read the moisture content. The result may not exceed the specification.

5.6.4.1 Construct a calibration curve with previously verified standards. Direct standard to unit with stainless steel lines which have been purged.

5.6.4.2 Obtain representative sample of gas to be analyzed and direct to unit as with the standards. (See NOTE 1.)

5.6.4.3 Construct a calibration curve and determine the ppm moisture content in sample gas. The result may not exceed the specification in Section 2 of this standard.

5.7 *Elemental Analysis in Deposited Layer* — Procedures for the analysis of elemental impurities in deposited layers of polysilicon should be designated and should be referenced from a recognized industry standard (including but not limited to ISO, ASTM, etc.).

5.7.1 *Typical Detection Limits*

<i>Compound</i>	<i>Detection Limit</i>
Carbon (C)	0.2 ppma
Oxygen (O)	0.2 ppma
Boron (B)	0.02 ppba
Aluminum (Al)	0.02 ppba
Gallium (Ga)	0.02 ppba
Phosphorus (P)	0.02 ppba
Arsenic (As)	0.02 ppba
Antimony (Sb)	0.02 ppba
Iron (Fe)	0.3 ppba
Chromium (Cr)	0.01 ppba
Nickel (Ni)	0.2 ppba
Copper (Cu)	0.02 ppba
Zinc (Zn)	0.03 ppba

5.8 *Resistivity* — Procedures for the measurement of resistivity in deposited layers of polysilicon should be designated and should be referenced from a recognized industry standard (including but not limited to ISO, ASTM, etc.).

NOTE 1: Before performing this or any other analytical operation it is necessary to perform the following:

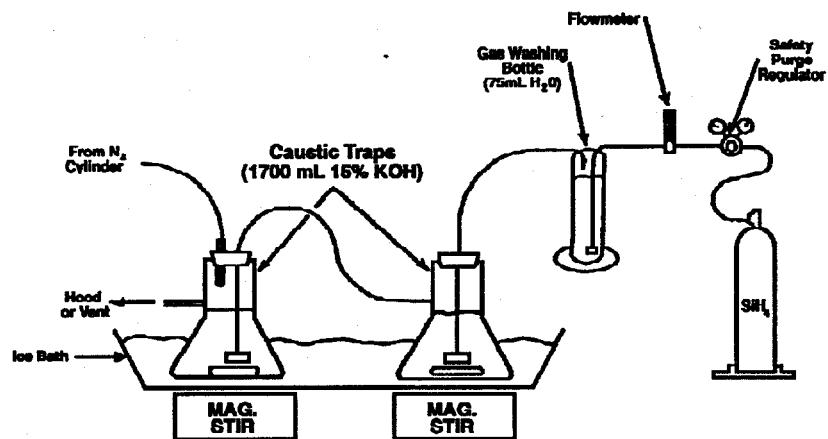
- Familiarize yourself with the products involved and the potential hazards by consulting the MSDS's for all hazardous substances involved and by consulting the CGA's "Handbook of Compressed Gases" for all gases involved.
- Know how to safely handle and store each hazardous gas by consulting the applicable CGA pamphlets.
- Use all necessary and applicable personal protective equipment.
- Follow all instructions relative to the safe operation of the equipment by consulting the LAC Safety Manual and applicable equipment manufacturers manual(s).

It must be noted that silane is a pyrophoric gas and all sample plumbing must be purged of air completely before introducing silane. Extra special attention must be paid to leak checking all connections and checking the vent plumbing to be certain that effluents are vented properly. If there are any

questionable connections or leaks, report this to the supervisor immediately and do not proceed with the analysis.

NOTE 2: Introduce the calibration standard as many times as necessary to achieve the desired precision. Detection limit

calculated on these calibrations should achieve at least that listed for this procedure.



Notes: 1. Direct Nitrogen into system at gentle rate.
2. Stirrers should turn at a moderate rate.

Figure 1
Chloride Scrubbing Apparatus

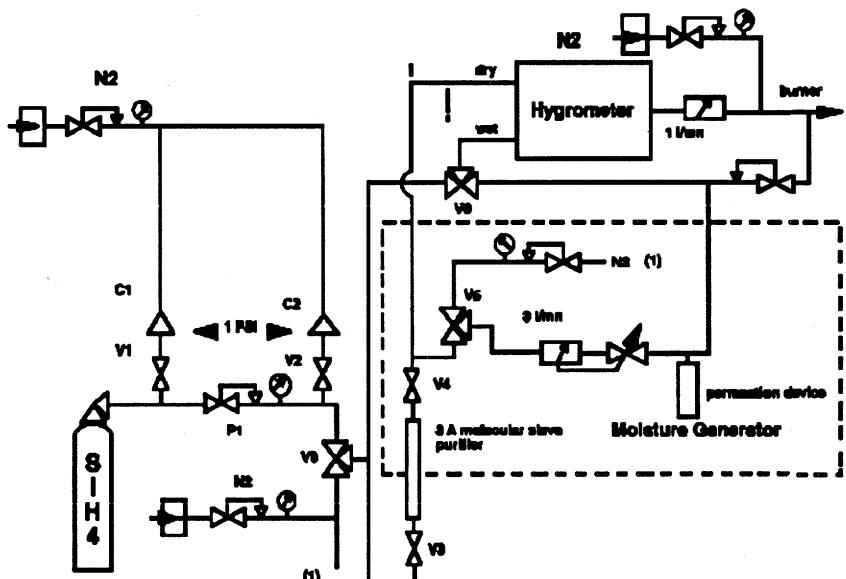


Figure 2
Moisture Analysis in Silane



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SEMI C3.24-0301

SPECIFICATION FOR SULFUR HEXAFLUORIDE (SF_6) IN CYLINDERS, 99.97% 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 November 22, 2000. Initially available at www.semi.org January 2001; to be published March 2001. Originally published in 1984; previously published in 1995.

1 Description

1.1 Sulfur hexafluoride is colorless and odorless. It is noncombustible and has a low toxicity. It is shipped as a liquefied gas under its own vapor pressure.

2 Specifications

QUALITY: 99.97%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm) (See NOTE 1.)</i>
Air	100
Carbon Tetrafluoride (CF_4)	100
Hydrogen Fluoride (HF)	1
Water (H_2O) (mol/mol)	8
TOTAL LISTED IMPURITIES	209

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

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	146.054	146.054
Boiling point at 1 atm	-63.7°C	-82.7°F
Density of gas at 20°C (68°F) and 1 atm	6.162 kg/m ³	0.385 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	5.114	5.114
Density of liquid at -50°C	1910 kg/m ³	119.2 lb/ft ³

4 Analytical Procedures (Notes 1, 2, and 3)

4.1 *Air and Carbon Tetrafluoride* — This procedure is for the determination of air and carbon tetrafluoride using a gas chromatograph with a thermal conductivity detector.

4.1.1 *Detection Limit* — 1 ppm (mol/mol)

4.1.2 *Instrument Parameters*

4.1.2.1 Column: 2.4 m (8 ft) by 6.4 mm (1/4 in) OD, 5.1 mm (0.201 in) ID stainless steel tubing packed with Porapak Q (80/100 mesh) or equivalent.

4.1.2.2 Carrier Flow: 30 mL/min helium m.

4.1.2.3 Sample Volume: 1 mL.

4.1.2.4 Temperatures:

Detector	110°C
Column	70°C

4.1.3 *Calibration Standards* — 90–100 ppm (mol/mol) carbon tetrafluoride, 90–110 ppm (mol/mol) Air (O_2/N_2 blend), balance helium.

4.1.4 *Operating Procedure*

4.1.4.1 Inject the calibration standard. Analyze the standard using the conditions described above. Record the retention times and peak areas.

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

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Calculate the concentrations of air and carbon tetrafluoride in the sample, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

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

4.2 *Hydrogen Fluoride* — This procedure is for the determination of hydrogen fluoride by quantitative collection in dilute sodium carbonate ($NaHCO_3$) and subsequent analysis by ion chromatography.

4.2.1 *Detection Limit* — 0.1 ppm for the method
Equipment Required:

500 mL Greenburgh impinger

50 mL Class A pipet

Type 316 stainless steel delivery system

5' Fluorocarbon tubing, 1/4 in OD by 0.03 in wall

Reagent Grade sodium bicarbonate

Deionized water

50 mL high-density polyethylene bottles
 Assorted Class A volumetric flasks
 Ion chromatograph
 Anion column which separates:
 F^- , Cl^- , Br^- , SO_4^{2-} , PO_4^{3-}

4.2.2 Ion Chromatograph Parameters:

Instrument:	Dionex 2000 (or equivalent)
Integrator:	Dionex 4270 (or equivalent)
Column:	HDIC AS4A (or equivalent)
Eluent:	0.001 M Na_2CO_3 + 0.001 M $NaHCO_3$
Flow Rate:	2 mL/minute
Pressure:	960 psia
Detector Conductivity Range:	3.0 microsiemens for less than 10 ppm
Sample:	100 microliters

4.2.3 Calibration — Calibrate the ion chromatograph by dissolving a weighed amount of ammonium fluoride (NH_4F) in deionized water and sequentially diluting it to a fluoride concentration of 0.1 ppmw in Class A volumetric flasks and analyzing it as specified by the instrument manufacturer.

4.2.4 Operating Procedure

4.2.4.1 Prepare a fresh solution of 1.7 mM sodium bicarbonate in deionized water.

4.2.4.2 Pipet 200 mL of the solution into a 500 mL Greenburgh impinger.

4.2.4.3 Bubble approximately 20 L of sulfur hexafluoride (SF_6) through the solution, using the delivery system shown in Figure 1.

4.2.4.4 Analyze the solution as specified by the instrument manufacturer.

4.2.4.5 Calculation — Calculate the concentration of hydrogen fluoride (HF) using the following formula:

$$\text{ppmw HFg} = \frac{(F^- \text{ ppmw}) \times Vg}{19.00 \times mg}$$

Where:

F^- ppm	=	Fluoride concentration in trapped solution.
Vg	=	Volume of collection solution (mL).
mg	=	Moles of SF_6 bubbled through the collecting solution.
19.00	=	Molecular weight of F^- .

The result may not exceed the specification in Section 2 of this Standard.

4.3 Water — This procedure is for the determination of trace moisture (water) in sulfur hexafluoride using a continuous flowing electrolytic hygrometer. (See Notes 4, and 5.)

4.3.1 Detection Limit — 1.0 ppm (vol/vol) or $-76^\circ C$ ($-105^\circ F$).

4.3.2 Sample Pressure and Flow — Set in accordance with instrument manufacturer's instructions.

4.3.3 Operation Check — Check the instrument periodically for correct operation. A gas containing a known amount of moisture should be passed through the instrument. Agreement between the hygrometer and the standard should be within their relative accuracies.

4.3.4 Operating Procedure

4.3.4.1 Obtain a continuous flow sample of sulfur hexafluoride source, using a clean, electropolished or passivated stainless steel line which has been purged dry after exposure to ambient moisture. (See Note 5.)

4.3.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and hygrometer until a stable reading is obtained. The reading may not exceed the specification in Section 2 of this Standard.

4.4 Notes

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 not contain more than 10% of the specified value of the component of interest, unless otherwise stated.

NOTE 3: Observe proper safety procedures for handling and disposing of sulfur hexafluoride (SF_6).

NOTE 4: The sampling system and hygrometer must be designed to operate at 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 hygrometer.

NOTE 5: A passivation procedure is described in Metals Handbook, Eighth Edition, Volume 2, ASM International, Metals Park, Ohio.

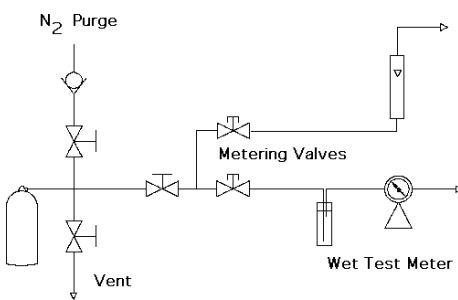


Figure 1



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SEMI C3.26-0301

SPECIFICATION FOR TUNGSTEN HEXAFLUORIDE (WF_6) IN CYLINDERS, 99.8% 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 November 22, 2000. Initially available at www.semi.org January 2001; to be published March 2001. Originally published in 1984; previously published in 1994.

1 Description

1.1 Tungsten hexafluoride is a colorless gas or colorless liquid. It is shipped as a liquefied gas under its own vapor pressure.

2 Specifications

QUALITY: 99.8%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm) (See NOTE 1.)</i>
Carbon Tetrafluoride (CF_4)	10
Hydrogen Fluoride (HF)	1000
Nitrogen (N_2)	50
Oxygen (O_2) + Argon (Ar)	50
Sulfur Hexafluoride	10
Silicon Tetrafluoride	10
TOTAL LISTED IMPURITIES	1130

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

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	297.85	297.85
Boiling point at 1 atm	17.1°C	62.8°F
Density of gas at 22.8°C (73°F) and 1 atm	12.9 kg/m ³	0.805 lb/ft ³
Specific gravity of gas	10.8	10.8
Density of liquid at boiling point	3440 kg/m ³	214.8 lb/ft ³

4 Analytical Procedures (See Notes 1, 2, 3, 4, 5, 6, 7)

4.1 *Nitrogen and Oxygen + Argon* — This procedure is for the determination of nitrogen and oxygen + argon in tungsten hexafluoride using a gas chromatograph with a thermal conductivity detector.

4.1.1 *Detection Limits* — 10 ppm (mol/mol) nitrogen, and 10 ppm (mol/mol) oxygen + argon.

4.1.2 Instrument Parameters

4.1.2.1 Columns: (See Figures 1, 2)

Column 1:	Porapak S, 80/100 mesh, 1.5 m (5 ft) by 6.4 mm (1/4 in) OD, 5.1 mm (0.2 in) ID, ss or equivalent.
Column 2:	Molecular sieve 5A, 80/100 mesh, 1.8 m (6 ft) by 4.8 mm (3/16 in) OD, 3.7 mm (0.147 in) ID, ss or equivalent.

4.1.2.2 Column Flow: 30 mL/min helium.

4.1.2.3 Sample Volume: 2 mL

4.1.2.4 Temperatures

Detector	70°C
Column	40°C

4.1.3 *Calibration Standard* — 50 ppm (mol/mol) nitrogen, 50 ppm (mol/mol) oxygen, balance helium.

4.1.4 Operating Procedures

4.1.4.1 Determine the times for valve switching and signal changes, and enter into the run table. An example of a run table follows.

<i>Time</i>	<i>Position</i>	<i>Function</i>
0 min.	1	Purge sample through loop. Backflush Porapak column. Connect MS column to the TCD.
1 min.	2	Inject sample onto Porapak column. Allow oxygen + argon and nitrogen to elute from the Porapak column to the MS column. Sequentially elute the oxygen + argon and nitrogen from the MS column to the TCD.
4 min.	1	Backflush Porapak column to vent for 8 minutes.

4.1.4.2 Set the valves in Position 1 (Figure 1).

4.1.4.3 Flow the calibration standard through the 10-port valve. Analyze standard using the conditions described above. Record retention times and peak areas.

4.1.4.4 Flow the sample to be tested through the 10-port valve for 1 minute. Analyze in the same manner as in 4.1.4.3.

4.1.4.5 The standard is again run as in 4.1.4.3.

4.1.4.6 Calculate the concentrations of nitrogen and oxygen + argon in the sample, using the formula below. The result may not exceed the specifications in Section 2 of this Standard.

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

4.2 Carbon Tetrafluoride, Hydrogen Fluoride, Silicon Tetrafluoride, and Sulfur Hexafluoride — This procedure is for the determination of these gases in tungsten hexafluoride using infrared spectrophotometry. (See Note 2.)

4.2.1 Detection Limits — 10 ppm (mol/mol) carbon tetrafluoride, 1000 ppm (mol/mol) hydrogen fluoride, 10 ppm (mol/mol) silicon tetrafluoride and 10 ppm (mol/mol) sulfur hexafluoride.

4.2.2 Instrument Parameters

4.2.2.1 Cell path length: 10 cm

4.2.2.2 Sample Cell Pressure: 1 atmosphere

4.2.2.3 Wavenumbers:

Component	Wavenumber (cm^{-1})
Carbon Tetrafluoride (CF_4)	1283
Hydrogen Fluoride (HF)	4076
Silicon Tetrafluoride (SiF_4)	1029
Sulfur Hexafluoride (SF_6)	948

4.2.3 Calibration Standards — 1% (mol/mol) hydrogen fluoride in tungsten hexafluoride, 100 ppm (mol/mol) silicon tetrafluoride in nitrogen, 25 ppm (mol/mol) each carbon tetrafluoride and sulfur hexafluoride in nitrogen.

4.2.4 Operating Procedures

4.2.4.1 Flow each calibration gas through the system for 1 minute. Pressurize the cell to 1 atmosphere. Record the absorbance of each calibration standard at the wavenumber listed in 4.2.2.3.

4.2.4.2 Flow the sample gas through the system for 1 minute. Pressurize the cell to 1 atmosphere. Record the absorbance at each of the appropriate wavenumbers. Calculate (See Note 1) the concentration of each gas, using the formula below. The results may not exceed the specification in Section 2 of this standard.

Measured Absorbance

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

4.3 Notes

NOTE 1: Tungsten hexafluoride interferes with the direct absorbance reading for silicon tetrafluoride. The absorbance for silicon tetrafluoride is determined by compensating for the interfering absorbance from the tungsten hexafluoride. Compensation is made by subtracting the absorbance contributed to this band by tungsten hexafluoride band at 930 cm^{-1} . This absorbance is halved and subtracted from the total peak absorbance at 1040 cm^{-1} , resulting in the peak absorbance for silicon tetrafluoride.

NOTE 2: Actual detection limits will be determined by the noise level and the resolution of the spectrometer. One way is to reduce the noise level is to use a Fourier Transform Infrared Spectrometer.

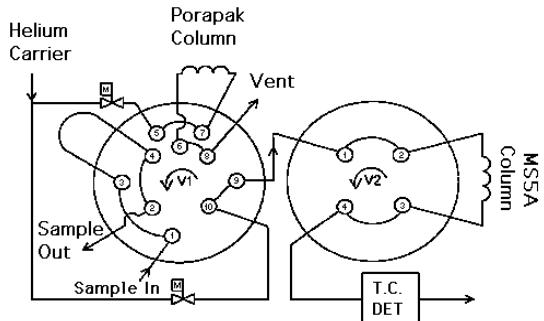
NOTE 3: All gases used in the analysis of the sample should not contain more than 10% of the specified value of the component of interest, unless otherwise stated.

NOTE 4: As tungsten hexafluoride has a low vapor pressure, the cylinder should be kept at room temperature (20°C) for at least 8 hours prior to analysis.

NOTE 5: Observe proper safety procedures for handling and disposing of tungsten hexafluoride.

NOTE 6: Prior to introducing tungsten hexafluoride purge the sample lines and instrument tubing with helium to remove moisture.

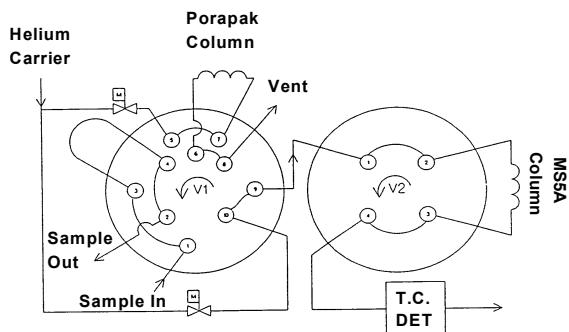
NOTE 7: Upon completing the analysis, cap off the manifold and instrument lines or purge the system with helium.



Position 1:

- Sample is purging through loop
- Porapak S column is backflushing
- MS5A column connected to detector

Figure 1



Position 2:

- V1 is turned
- Sample flows through sample loop to the Porapak S column and then to MS5A column

Figure 2

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SEMI C3.52-0200

STANDARD FOR TUNGSTEN HEXAFLUORIDE, 99.996% QUALITY

This standard 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 15, 1999. Initially available at www.semi.org February 2000, to be published February 2000. Originally published in 1995; previously published in 1996.

1 Purpose

1.1 To define the specification and analytical methods and validate the specifications for WF₆.

2 Scope

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

3 Description

3.1 Tungsten hexafluoride is a colorless, odorless gas or a clear liquid.

4 Specifications

Quality: 99.996%

<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Oxygen (O ₂) & Argon (Ar)	1 ppm
Nitrogen (N ₂)	5 ppm
Carbon Dioxide (CO ₂)	1 ppm
Carbon Tetrafluoride (CF ₄)	1 ppm
Hydrogen Fluoride (HF)	20 ppm
Silicon Tetrafluoride (SiF ₄)	1 ppm
Sulfur Hexafluoride (SF ₆)	1 ppm
Carbon Monoxide (CO)	1 ppm
TOTAL SPECIFIED IMPURITIES	31 ppm

<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Cobalt (Co) & Manganese (Mn)	0.01 ppm w
Lead (Pb)	0.02 ppm w
Chromium (Cr), Zinc (Zn), & Uranium (U)	0.05 ppm w
Calcium (Ca) & Magnesium (Mg)	0.10 ppm w
Nickel (Ni), Copper (Cu), Sodium (Na), & Potassium (K)	0.15 ppm w
Iron (Fe)	0.02 ppm w
Thorium (Th)	0.1 ppm w
Molybdenum (Mo)	1 ppm w

5 Physical Constants

	<i>Metric Units</i>	<i>U. S. Units</i>
Molecular weight	297.85	297.85
Boiling point	17.1°C	62.8°F
Density of gas at 22.8°C (73°F) and 1 atm	12.9 kg/m ³	0.805 lb/ft ³
Specific gravity of gas	10.8	10.8
Density of liquid at boiling point	3440 kg/m ³	

6 Analytical Procedures (see NOTE 1)

6.1 *Carbon Tetrafluoride, Silicon Tetrafluoride, Carbon Dioxide, and Sulfur Hexafluoride* — This procedure is for the determination of carbon tetrafluoride, silicon tetrafluoride, carbon dioxide, and sulfur hexafluoride in tungsten hexafluoride using a gas chromatograph equipped with a helium ionization detector. A backflush is used in order to protect the detector from the main component.

6.1.1 *Detection Limits* — 0.3 ppm (mole/mole) carbon tetrafluoride, 1 ppm (mole/mole) silicon tetrafluoride, 0.1 ppm (mole/mole) carbon dioxide, and 0.5 ppm (mole/mole) sulfur hexafluoride.

6.1.2 Instrument Parameters

6.1.2.1 Columns:

Column 1:	10% Kel F Nr10 on Chromosorb T, 4.0 m (13 ft) by 3.2 mm (1/8 in) nickel or equivalent,
Column 2:	Preconditioned HAYESEP Q, 60/80 mesh, 3 m (10 ft) by 3.2 mm (1/8 in) or equivalent. The column should be treated by repeated WF ₆ sample injections at 125°C.

6.1.2.2 *Carrier Flow* — 26 mL/min helium, 6.0 grade.

6.1.2.3 Temperatures:

Detector	50°C
Column Oven	50°C

6.1.2.4 *Sample Volume* — 2 mL

6.1.2.5 *Calibration Standards* — 1 and 5 ppm (mole/mole) carbon tetrafluoride, carbon dioxide, and sulfur hexafluoride in helium (see Figures 1 and 2).

10 ppm silicon tetrafluoride in helium or permeation device (see Figure 3).

6.1.3 Operating Procedure

6.1.3.1 Attach a suitable pressure regulator to the standard cylinders. Connect the regulator to the dedicated WF₆ handling system which is connected to the chromatographic sampling valve.

6.1.3.2 Purge the sampling lines with the standard for at least one minute.

6.1.3.3 Inject the standard into the gas chromatograph. Record the retention times and peak areas. Order of elution for the above mentioned standard is carbon tetrafluoride, silicon tetrafluoride, carbon dioxide, and sulfur hexafluoride.

6.1.3.4 Inject the WF₆ sample to be analyzed in the same manner as the calibration standard. Record the retention times and peak areas.

6.1.3.5 Compare the average peak areas of the calibration standard to that of the detected peak in tungsten hexafluoride. Calculate the concentration of each impurity using the standard and sample peak areas and the standard concentration. The results may not exceed the specifications.

6.2 Oxygen and Argon, Nitrogen, and Carbon Monoxide — This procedure is for the determination of oxygen and argon, nitrogen, and carbon monoxide in tungsten hexafluoride using a gas chromatograph equipped with a helium ionization detector. A backflush is used in order to protect the detector from the main component.

6.2.1 Detection Limits

6.2.2 Instrument Parameters

6.2.2.1 Columns:

Column 1:	10% Kel F Nr 10 on Chromosorb T, 4.0 m (13 ft) by 3.2 mm (1/8 in) nickel or equivalent.
Column 2:	Preconditioned HAYESEP Q, 60/80 mesh, 3 m (10 ft) by 3.2 mm (1/8 in) or equivalent. The columns should be treated by repeated WF ₆ sample injections at 125°C.
Column 3:	Molecular sieve 5 Å, 60/80 mesh, 2 m (6 ft) by 3.2 mm (1/8 in).

6.2.2.2 Carrier Flow — 26 mL/min helium, 6.0 grade.

6.2.2.3 Temperatures:

Detector	50°C
Column Oven	50°C

6.2.2.4 Sample Volume — 2 mL

6.2.2.5 Calibration Standards — 1 and 5 ppm (mole/mole) oxygen, nitrogen, and carbon monoxide in helium (see Figure 4).

6.2.3 Operating Procedure

6.2.3.1 Attach a suitable pressure regulator to the standard cylinder. Connect the regulator to the dedicated WF₆ handling system, which is connected to the chromatographic sampling valve.

6.2.3.2 Purge the sampling lines with the standard for at least one minute.

6.2.3.3 Inject the standard into the gas chromatograph. Switch the valves in order to operate in the same conditions as the WF₆ sample analysis. Record the retention times and peak areas. Order of elution for the above mentioned standard is oxygen and argon, nitrogen, and carbon monoxide.

6.2.3.4 Inject the WF₆ sample in the same manner as the calibration standard. Record the retention times and peak areas.

6.2.3.5 Compare the average peak areas of the calibration standard to that of the detected peak in the tungsten hexafluoride. Calculate the concentration of each impurity using the standard and sample areas and the standard concentration. The results may not exceed the specifications.

6.3 Hydrogen Fluoride — This procedure is for the determination of hydrogen fluoride in tungsten hexafluoride using Fourier Transform Infra Red (FTIR) analyzer.

6.3.1 Detection Limit — 0.5 ppm (mole/mole).

6.3.2 Instrument Parameters

- cell with CaF₂ windows
- HF band: 4038,8 cm⁻¹
- resolution: 2 cm⁻¹
- path length: 12 cm

6.3.3 Calibration Standard — Calibrate with low vapor pressure hydrogen fluoride or permeation device.

6.3.4 Operating Procedure — Carefully dry the cell before sampling tungsten hexafluoride. Then purge it with tungsten hexafluoride and record the concentration. When it is steady, that means the line is purged and the true hydrogen fluoride concentration is obtained.

6.4 Elemental Impurities — This procedure is for the determination of Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Th, U, and Zn in the nonvolatile residue of liquid phases tungsten hexafluoride (WF₆) and analyzed

by Inductively Coupled Plasma Mass Spectrometry (ICPMS).

6.4.1 Detection Limits (ppb wt/wt) — Ca: 1.5; Co: 0.1; Cr: 0.8; Cu: 0.5; Fe: 1; K: 1.5; Mg: 0.1; Mn: 0.1; Mo: 0.5; Na: 0.6; Ni: 1; Pb: 0.1; Th, U: 0.01; and Zn: 0.8.

6.4.2 Sampling Apparatus — Figure 5 shows the sampling apparatus used for obtaining a liquid phase sample of WF₆. The sampling apparatus consists of:

- Cylinder Inverter,
- PFA Vessels,
- PFA Valves, and
- PFA Tubing.

The whole sampling system is placed inside a hood.

6.4.3 Water — At least 18 MΩ DI water should be used for all dilutions, blanks, and washing.

6.4.4 Reagents — All reagents are of the highest purity available minimizing background metal contamination. The following reagents are needed to prepare the sample and standards:

- Aqueous Ammonia,
- Aqueous HNO₃,
- Aqueous HF, and
- NIST traceable ICP standards.

6.4.5 Sampling Liquid Tungsten Hexafluoride

6.4.5.1 Place about 400 ml of DI water in each of the hydrolysis vessels. Obtain the weight of each of the hydrolysis vessels.

6.4.5.2 Assemble the apparatus as shown in Figure 1. Do not tighten the cylinder CGA connection. All PFA parts should be clean and dried prior to assembly.

6.4.5.3 Open valves V2, V3, V4 and V5. Set the house nitrogen pressure to approximately 15 psig. Purge the system with nitrogen for about 15 minutes. **TIGHTEN THE CYLINDER CGA CONNECTION.**

6.4.5.4 Close V2. The system should now be under pressure. Check for leaks and repair if necessary.

6.4.5.5 Open V1 and evacuate the system for approximately 10 minutes.

6.4.5.6 Close all valves.

6.4.5.7 Fill sample vessel by opening cylinder valve and valve V5. Fill the sample vessel approximately 2/3 full. Close cylinder valve.

6.4.5.8 Close V5. Open V2, V4, and V6. This will carry the WF₆ vapors to the hydrolysis vessel.

6.4.5.9 Purge the system with nitrogen for a few minutes after the WF₆ liquid in the sample cup has totally evaporated.

6.4.5.10 Close all valves. Remove the hydrolysis vessels and weigh the vessels.

6.4.5.11 The weight gain in the hydrolysis vessel will give the weight of the sampled WF₆. Typically the weight gain of the second and third vessels will be less than 1% of the weight gain of the first hydrolysis vessel.

6.4.5.12 Remove the sample vessel and dissolve the residue.

6.4.6 Sample Preparation

6.4.6.1 Add 20 ml DI water to a 100 ml volumetric flask.

6.4.6.2 Open the sample cup and add 5 ml of aqueous ammonia to the cup.

6.4.6.3 Swirl in the cup and place in a 100 ml volumetric flask.

6.4.6.4 Add 5 ml Nitric Acid to the cup.

6.4.6.5 Swirl and place in the same 100 ml volumetric flask.

6.4.6.6 Add 5 ml Hydrofluoric Acid to the cup.

6.4.6.7 Swirl and place in the same 100 ml volumetric flask as before.

6.4.6.8 Dilute the solution with DI water up to the mark of the 100 ml volumetric flask.

6.4.6.9 Analyze for metals in the sample.

6.4.7 ICPMS Analysis Procedure

6.4.7.1 This procedure is used to analyze for trace Na, K, Cr, Fe, Th and U using multi point calibration. Other elements can be analyzed using a single point calibration. Elements for which stable standards can not be obtained, can be analyzed using interpolation and a knowledge of the instrument response function. The ICPMS should be calibrated using matrix matched multielement standards. At least one spiked sample should be analyzed as part of the analytical procedure. The recovery of the spikes should be within $\pm 25\%$. At least three standards and a blank should be used for the calibration. Regression based approach should be used to calculate the response factors. The limit of detection (LOD) should be calculated following SEMI C10, Guide for Determination of Method Detection Limits.

6.4.7.2 The concentration of impurities in gas phase WF_6 is given by

$$\text{Conc. in } \text{WF}_6 = \frac{\text{Conc. in Solution} \times \text{Wt. of Solution}}{\text{Weight of Sampled } \text{WF}_6}$$

NOTE 1: All gases used in the analysis of the sample should contain not more than 10% of the specified value of the component of interest unless otherwise specified.

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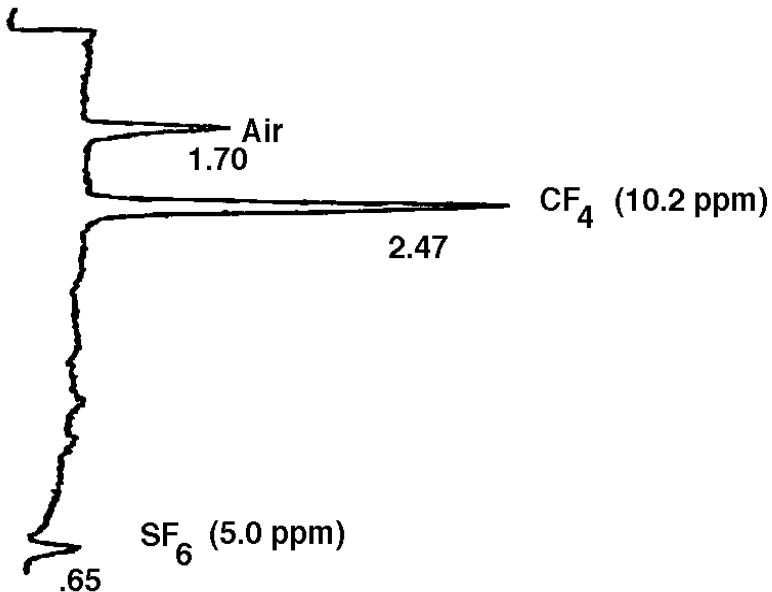


Figure 1
CF₄ and SF₆ Standards in He

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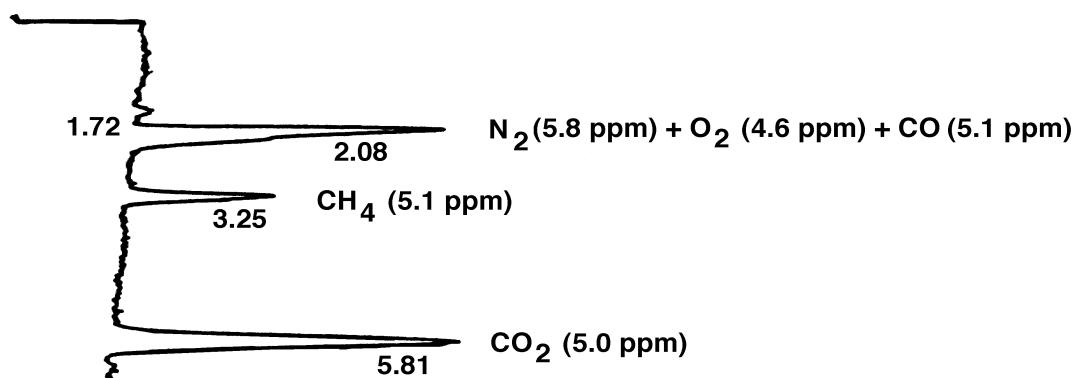


Figure 2
CO₂ Standards in He

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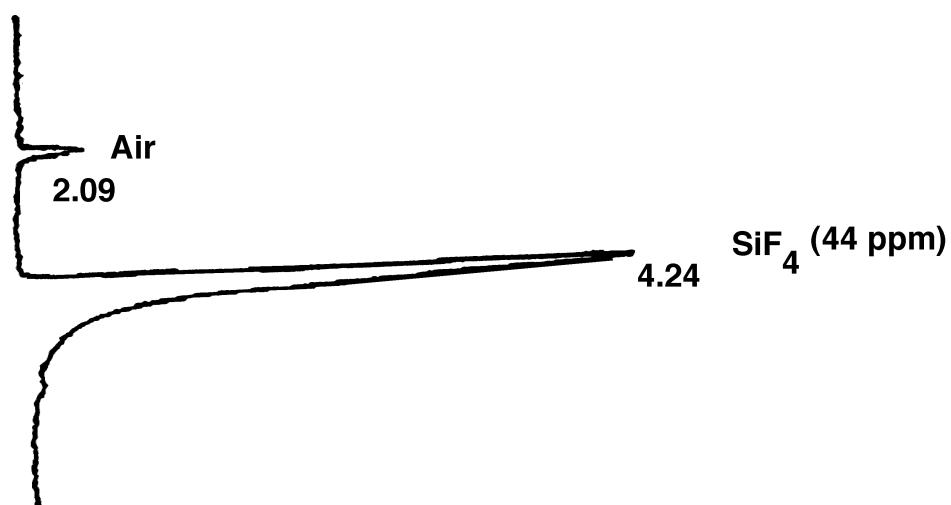


Figure 3
SiF₄ Standard in He

CHANNEL A INJECT

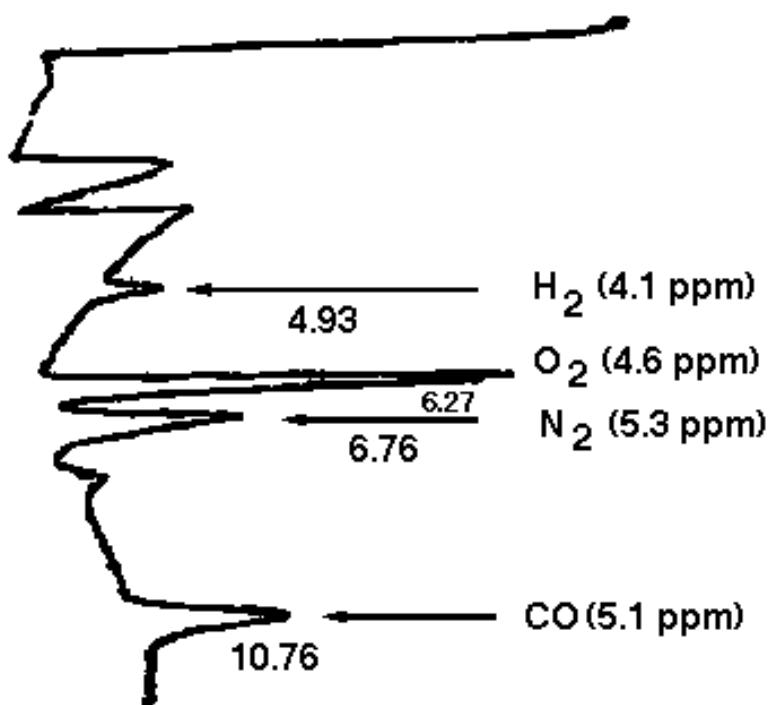


Figure 4
O₂, N₂, and CO Standards in He

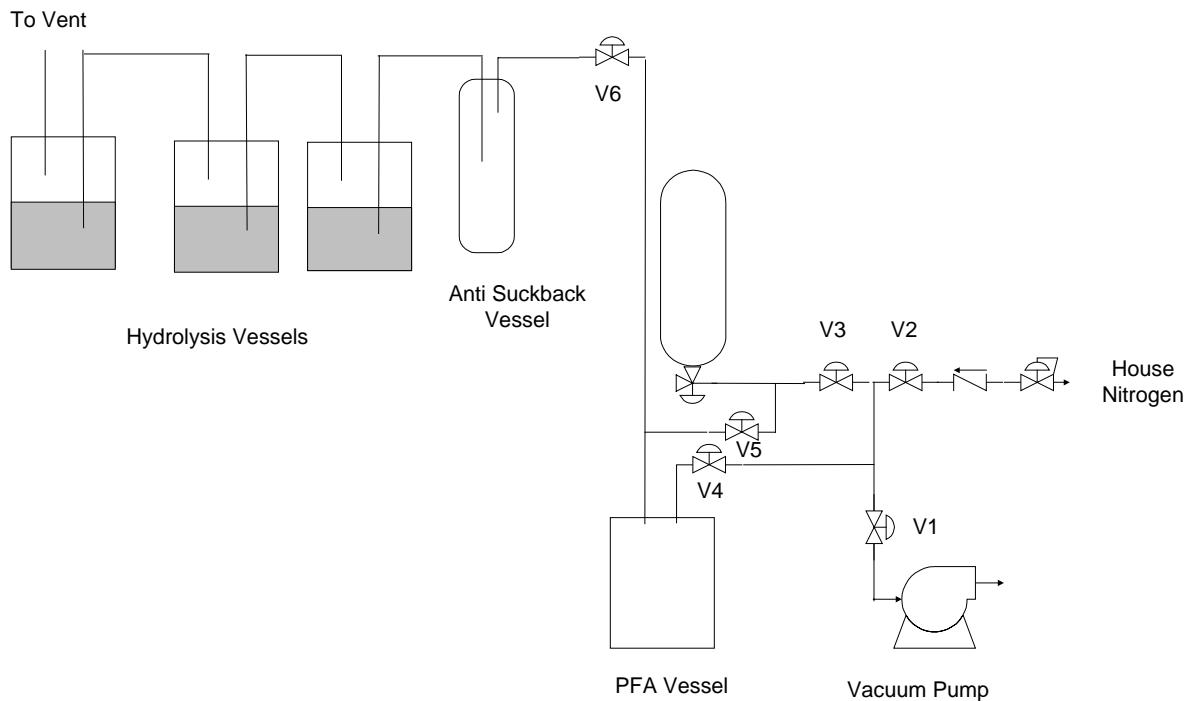


Figure 5
Sampling System for WF_6

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SEMI C6.3-89 (Reapproved 0303)

PARTICLE SPECIFICATION FOR GRADE 20/0.2 HYDROGEN (H₂)

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 October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published in 1989.

1 Purpose

1.1 The purposes of this document are (1) to set a maximum permissible particle concentration for Grade 20/0.2 hydrogen gas and (2) to describe a reference method for its verification.

2 Scope

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

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

3 Referenced Standards

None.

4 Terminology

4.1 Definitions

4.1.1 *background count* — The total number of counts registered by a specific particle counter within the time needed to sample 1.0 SCF of gas under conditions where zero particles transverse the sensing volume, averaged over at least a sequence of twenty-four consecutive sampling periods of 1.0 SCF each or eight consecutive periods of 30 minutes each, whichever is longer. The background count is to be reported as mean number of count per SCF; the number of hours on which the average is based is also reported.

4.1.2 *gas sample volume* — The volume of the sample, expressed in standard cubic feet (SCF) is the volume occupied by the gas sample at standard conditions, 20° C (68° F) and 1.00 atmosphere pressure.

4.1.3 *particle concentration* — The total number of particles counted, divided by the total gas sample volume, and corrected for background count as defined in Section 4.1.1, rounded to the nearest integer, for particles equal to or larger than the specified size.

4.1.4 *particle size/particle diameter* — The optical equivalent diameter as detected by a given light scattering particle counter.

NOTE 1: The optical equivalent diameter varies from counter to counter and with differing particle shape and refractive index.

4.1.5 *sampling period* — The time needed to sample 1.0 SCF or 30 minutes, whichever is longer.

5 Apparatus

5.1 *Light Scattering Optical Particle Counter* — With a lower detection limit of 0.2 micrometers or less, based on a calibration with polystyrene latex spheres; and with a maximum background of 2 counts/SCF at 0.2 micrometers. It should be suitable for use in hydrogen service per the manufacturer's specifications or encased in an explosion-proof enclosure which satisfies Class I, Division I of the NFPA Code.

6 Test Method

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

6.1 The sampling point should be as close as practical to the downstream side of the supplier's final gas filter. Sampling lines should be as short as possible.

6.2 Suggested sampling probe configuration for turbulent main line flow:

Alternative a:	Figure 1a (preferred)
Alternative b:	Figure 1b
Alternative c:	Figure 1c
Alternative d:	Figure 1d

6.3 For configurations 1a, 1b, and 1c, 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. Sampling tube ID should be no less than 2 mm (0.08 in).

6.4 Determine the background count for the particle counter by using the standard method recommended by the instrument manufacturer using hydrogen gas.

7 Specification

7.1 Maximum permissible total concentration: 20 particles per SCF equal to or larger than 0.2 micrometers, after subtracting background count as defined in Section 4.1.1.

7.2 The specification will be considered as met, if the particle concentration does not exceed 20 particles per SCF in any five consecutive sampling periods as defined in Section 4.1.5.

8 Report

8.1 Report each of the five consecutive sampling periods to read as total number of particles per SCF₇ of gas, equal to or larger than 0.2 micrometers optical equivalent diameter, without reference to any specific size distribution function. The background count shall be reported as defined in Section 4.1.1.

9 Calibration

9.1 The optical particle counter is normally calibrated using latex spheres of a known diameter suspended in a gas. Since the gas density and refractive index enter into the amount of scattering detected by the instrument, hydrogen should be used for calibration.

10 Safety

10.1 The lower explosive limit (LEL) for hydrogen in air is 4%; therefore, proper precautions should be taken to insure that the maximum possible concentration of hydrogen in air does not exceed 2%. The particle counter and any other ancillary equipment must be checked for leaks and the hydrogen exiting the equipment must be disposed of appropriately to insure that its concentration in air at any point does not exceed 2%.

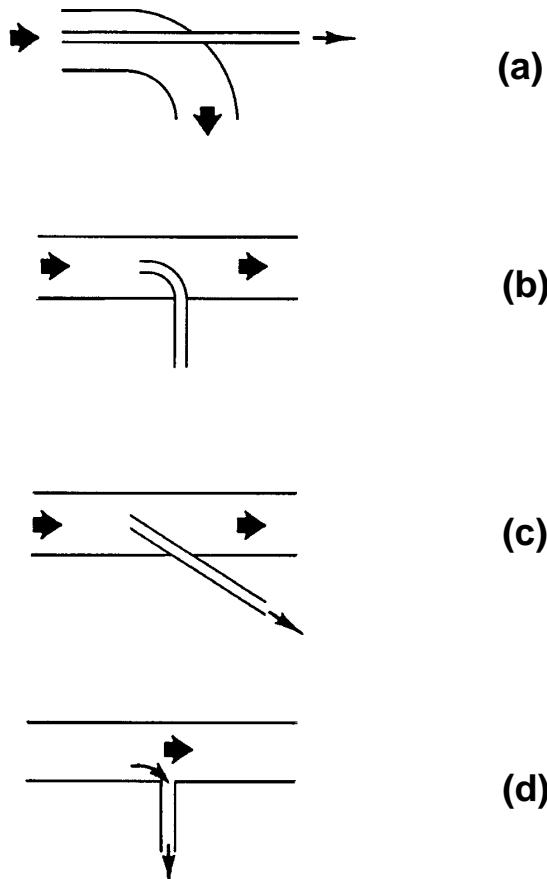


Figure 1
Schematic Diagrams of Configurations of
Different Merit for Obtaining Particle Samples
from Pipelines

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SEMI C6.4-90 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 20/0.02 NITROGEN (N₂) AND ARGON (Ar) 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 1990.

1 Purpose

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

2 Scope

2.1 This document applies only to nitrogen gas and argon gas delivered through pipelines; it is not applicable to cylinder gases or gases in their 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
̄X _M =	Average observed concentration of counts in the pipeline gas sample
̄X _B =	Average observed concentration of background counts
̄X _C =	Calculated concentration of particles in the pipeline gas
S _M =	Standard deviation of ̄X _M
S _B =	Standard deviation of ̄X _B
S _C =	Standard deviation of ̄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 (̄X_M, ̄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 (̄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.:

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

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

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

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

4 Apparatus

4.1 *Particle Counter* — An instrument suitable for counting particles in gaseous nitrogen or argon with a minimum detection efficiency of 90% 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 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 0.02 micrometers or more 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 S_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 deviations does not exceed 20 particles per 25 standard liters, i.e.:

$$\bar{X}_C + 2^* S_C \leq 20 \text{ particles/25 standard}$$

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.

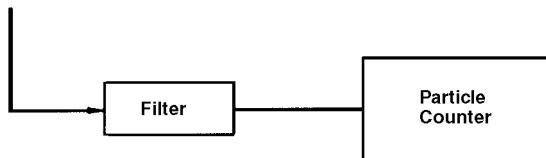


Figure 1
Suggested Assembly for Determining Particle Counter Background

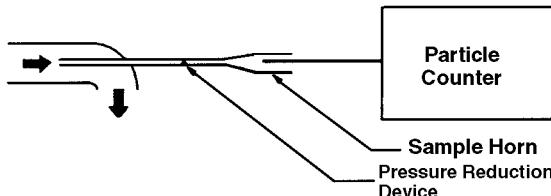


Figure 2
Schematic Diagram of Configuration for Obtaining Particle Samples from Pipelines

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SEMI C6.5-90 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 NITROGEN (N₂) AND ARGON (Ar) 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 1990.

1 Purpose

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

2 Scope

2.1 This document applies only to nitrogen gas and argon gas delivered through pipelines; it is not applicable to cylinder gases or gases in their 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
̄X _M =	Average observed concentration of counts in the pipeline gas sample
̄X _B =	Average observed concentration of background counts
̄X _C =	Calculated concentration of particles in the pipeline gas
S _M =	Standard deviation of ̄X _M
S _B =	Standard deviation of ̄X _B
S _C =	Standard deviation of ̄X _C

3.2 *Particle Size/Particle Diameter* — The optical equivalent diameter as detected by a given light-scattering particle counter.

3.3 *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.

3.4 *Average Observed Concentration of Counts (̄X_M, ̄X_B)* — The average concentration of counts, i.e.:

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

3.5 *Calculated Concentration of Particles (̄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.:

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

3.6 *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.:

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

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

4 Apparatus

4.1 *Particle Counter* — An instrument suitable for counting particles in gaseous nitrogen or argon with a lower detection limit of 0.2 micrometers or less, based on calibration with polystyrene latex spheres.

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 0.2 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 the time to sample 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 the time to sample 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.

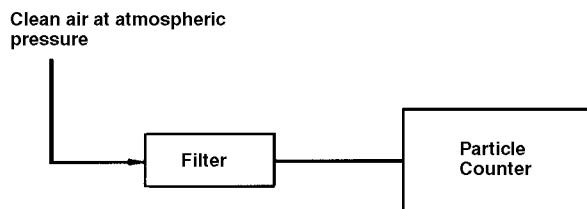


Figure 1
Suggested Assembly for Determining Particle Counter Background

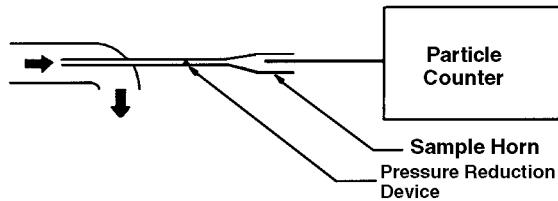


Figure 2
Schematic Diagrams of Configurations of Different Merit for Obtaining Particle Samples from Pipelines



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SEMI C6.6-90 (Reapproved 1102)

PARTICLE SPECIFICATION FOR GRADE 10/0.1 NITROGEN (N₂) AND ARGON (Ar) 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 1990.

1 Purpose

1.1 The purposes of this document are: (1) to set a maximum permissible particle concentration for 10/0.1 grade nitrogen and argon bulk supply gases, and (2) to describe a reference method for its verification.

2 Scope

2.1 This document applies only to nitrogen gas and argon gas delivered through pipelines; it is not applicable to cylinder gases or gases in their 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 ith 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
̄X _M =	Average observed concentration of counts in the pipeline gas sample
̄X _B =	Average observed concentration of background counts
̄X _C =	Calculated concentration of particles in the pipeline gas
S _M =	Standard deviation of ̄X _M
S _B =	Standard deviation of ̄X _B
S _C =	Standard deviation of ̄X _C

3.2 *Particle Size/Particle Diameter* — The optical equivalent diameter as detected by a given light-scattering particle counter.

3.3 *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.

3.4 *Average Observed Concentration of Counts (̄X_M, ̄X_B)* — The average concentration of counts, i.e.:

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

3.5 *Calculated Concentration of Particles (̄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.:

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

3.6 *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.

4 Apparatus

4.1 *Particle Counter* — An instrument suitable for counting particles in gaseous nitrogen or argon with a lower detection limit of 0.1 micrometers or less, based on calibration with polystyrene latex spheres.