

9.2.3 Using purified nitrogen as the purge gas, complete one purge cycle with the purge gas pressure at 522 kPa (g) [80 psi (g)], vacuum at -75 kPa (g) [-22" Hg (g)], and dwell times for pressurization and vacuum evacuation at 5 and 15 seconds, respectively.

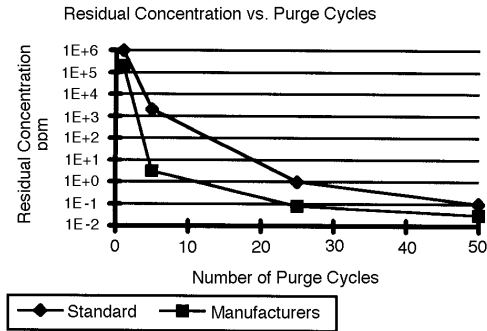


Figure 2
Data Presentation for Purge Efficacy — Non-Interactive Gas

9.2.4 Flow purified nitrogen using a pigtail bleed at 1 slpm or at the oxygen analyzer manufacturer's recommended flow rate, whichever is higher, into the oxygen analyzer for 10 minutes or until below the detection limit of the oxygen analyzer.

9.2.5 Record the pressure and vacuum measured with the transducer and the peak oxygen concentration as in Table 1. It is highly recommended that, in addition to the peak oxygen concentration, the oxygen concentration readings also be recorded at regular intervals during the measurement, so that a graph of the oxygen concentration level in the pigtail bleed gas versus time can be drawn if necessary.

Table 1 Data Collection Form

	<i>Manufacturer's Recommended Purge Parameters</i>	<i>SEMI Standard Purge Parameters</i>
Pressure		kPa (g)
Vacuum		kPa (g)
Cycles	Peak Residual Oxygen	
1		
5		
25		
50		

9.2.6 Repeat Sections 9.2.2–9.2.5 process for 2, 5, 25, and 50 cycles. Additional tests using other purge cycle numbers are encouraged and may provide a more

complete characterization of the purge performance of the test piece.

9.2.7 Plot the peak residual oxygen concentration versus number of cycles as in Figure 2 for standard pressures, vacuum, and dwell time.

9.3 Purge efficacy determination with interactive gas at manufacturer's pressure, vacuum, and dwell times.

9.3.1 Connect gas source equipment as in Figure 1.

9.3.2 To challenge the gas panel with a known contaminant gas, flow wet (2 ppm) N₂ at 2 slpm and 138 kPa (g) [20 psi (g)] for 30 minutes or until the process outlet concentration reaches the inlet concentration, whichever is less.

9.3.3 Using purified nitrogen as the purge gas, complete one purge cycle at manufacturer's recommended pressure, vacuum, and dwell times.

9.3.4 Flow purified nitrogen using a pigtail bleed at 1 slpm or at the moisture analyzer manufacturer's recommended flow rate, whichever is higher, into the moisture analyzer for 30 minutes or until below the detection limit of the moisture analyzer.

9.3.5 Record the pressure and vacuum measured with the pressure transducer and peak residual moisture concentration in Table 2. It is highly recommended that, in addition to the peak moisture concentration, the moisture concentration readings also be recorded at regular intervals during the measurement, so that a graph of the moisture concentration level in the pigtail bleed gas versus time can be drawn if necessary.

9.3.6 Repeat Sections 9.3.2–9.3.5 process for 2, 3, 4, 5, 10, 25, and 50 cycles. Additional tests using other purge cycle numbers are encouraged and may provide a more complete characterization of the purge performance of the test piece.

9.3.7 Plot the peak residual moisture concentration versus number of cycles as in Figure 3 for manufacturer's pressure, vacuum, and dwell time.

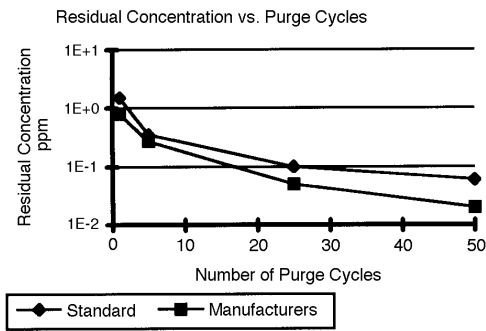


Figure 3
Data Presentation for Purge Efficacy
Interactive Gas

9.4 Purge efficacy determination with interactive gas at standard pressure, vacuum, and dwell time.

9.4.1 Connect gas source equipment as in Figure 1.

9.4.2 To challenge the gas panel with a known contaminant gas, flow wet (2 ppm) N₂ at 2 slpm and 138 kPa (g) [20 psi (g)] for 30 minutes or until the process outlet concentration reaches the inlet concentration, whichever is less.

9.4.3 Using purified nitrogen as the purge gas, complete one purge cycle with the purge gas pressure at 522 kPa (g) [80 psi (g)], vacuum at -75 kPa (g) [-22" Hg (g)], and dwell times for pressurization and vacuum evacuation at 5 and 15 seconds, respectively.

9.4.4 Flow purified nitrogen using a pigtail bleed at 1 slpm or at the moisture analyzer manufacturer's recommended flow rate, whichever is higher, into the moisture analyzer for 30 minutes or until below the detection limit of the moisture analyzer.

9.4.5 Record the pressure and vacuum measured with the pressure transducer and peak residual moisture concentration in Table 2. It is highly recommended that, in addition to the peak moisture concentration, the moisture concentration readings also be recorded at regular intervals during the measurement, so that a graph of the moisture concentration level in the pigtail bleed gas versus time can be drawn if necessary.

9.4.6 Repeat Sections 9.4.2–9.4.5 process for 2, 3, 4, 5, 10, 25, and 50 cycles. Additional tests using other purge cycle numbers are encouraged and may provide a more complete characterization of the purge performance of the test piece.

9.4.7 Plot the peak residual moisture concentration versus number of cycles as in Figure 4 for standard pressure, vacuum, and dwell time.

Table 2 Data Collection Form

	<i>Manufacturer's Recommended Purge Parameters</i>	<i>SEMI Standard Purge Parameters</i>
Pressure		kPa (g)
Vacuum		kPa (g)
Cycles	Peak Residual Moisture	
1		
5		
25		
50		

10 Certification

10.1 When specified in the purchase order or contract, the manufacturer's or supplier's certification shall be furnished to the purchaser stating that the articles furnished have been tested in accordance with this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

11 Related Documents

11.1 *SEMI Standard*

SEMI F13 — Guide for Gas Source Control Equipment

11.2 *Compressed Gas Association Document¹*

CGA Pamphlet P1 — Safe Handling of Compressed Gases

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 mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

¹ Compressed Gas Association, Inc., 1725 Jefferson Davis Highway, Suite 1004, Arlington, VA 22202

SEMI F30-0298

START-UP AND VERIFICATION OF PURIFIER PERFORMANCE

TESTING FOR TRACE GAS IMPURITIES AND PARTICLES AT AN INSTALLATION SITE

1 Purpose

1.1 The purpose of this procedure is to verify the performance of purifiers by employing analytical instrumentation to measure gas impurities and particles to customer specifications. If specific inlet challenge(s) and/or inlet measurements are required, it should be discussed beforehand with the customer. Inlet impurities must be measured by part-per-million (PPM) or part-per-billion (PPB) analytical equipment. This procedure applies only to large scale bulk purifiers rated at greater than 50 liters-per-minute (LPM) flowrate.

2 Scope

2.1 Verify performance of large scale purifiers in nitrogen, argon, helium, oxygen, and hydrogen service. Verification tests are done at PPB or sub-PPB levels of gaseous impurities and sub-micron sizes of particles measured downstream of any installed filter modules. Tests are done at maximum achievable flow of purifier, and/or customer's specified percentages of maximum flow.

3 Limitations

3.1 PPB and sub-PPB gaseous impurity levels are achievable using atmospheric pressure ionization mass spectrometry (APIMS), which is the preferred method of choice, and the reduction gas detector (RGD)-gas chromatograph. APIMS is currently not available for oxygen service. A partial list of non-APIMS measuring equipment for use in oxygen service is in Appendix 1 for commonly measured impurities.

4 Referenced Documents

- 4.1 Approved procedures for operation of analytical equipment.
- 4.2 Approved gas sampling and purifier procedures.

5 Terminology

None.

6 Summary of Method

6.1 The purifier is started, and the operation is checked. Each purifier bed is regenerated, and analytical tests are done. The analytical results

determine if the purified gas meets the customer specifications.

7 Interferences

7.1 The following sources might contribute to misleading or high analytical results. Some identified sources are unpurged, dead-ended piping or isolation valves, leaks in gas distribution system, insufficient purge flow, and insufficient system clean-up time.

8 Apparatus

8.1 Face seal fitting(s) with metal gaskets and stainless steel tubing for sampling.

8.2 Dynamic dilution system for diluting calibration standards to PPB and sub-PPB levels for calibration of analytical equipment.

8.3 *APIMS* — The sample gas, nitrogen, is introduced into an APIMS where a small amount of it is ionized. By collision with ionized nitrogen, impurity molecules are ionized with high efficiency. The mass analyzer, which can be a quadrupole, time-of-flight (TOF), or even a magnetic sector, separates and focusses the ions by their mass-to-charge ratio. An electron multiplier detects and counts each ion fragment and amount.

8.4 *Reduction Gas Detector-Gas Chromatograph (RGD-GC)* — The reduction gas detector is a heated mercuric oxide bed that reacts with reducing gases, such as hydrogen and carbon monoxide. The mercury evolved is detected and displayed as a peak. The GC employs a heated molecular sieve column to separate the H₂ and CO.

8.5 *Ultratrace Analytical Instrumentation Required other than APIMS* — Ultratrace instrumentation is defined as having sufficient sensitivity to measure all impurities of interest at the specified level of the customer at the PPB or sub-PPB.

8.6 Particle counter.

8.7 Data collection and reduction system.

9 Reagents and Materials

9.1 Certified calibration standards.

10 Safety Precautions

10.1 The testing area should have adequate room ventilation and atmospheric monitors.

10.2 Instruments should be exhausted to vent, and if required in Class 1 environments, should be case-purged or in an approved enclosure.

10.3 Testing personnel should be aware of customer alarm and evacuation procedures.

10.4 Designated customer contact required during testing.

11 Sampling

11.1 Test each purifier bed independently to verify performance to customer specifications. Refer to gas supplier's certificate of conformance for inlet impurity levels. Refer to Figure 1 for overall test sequence.

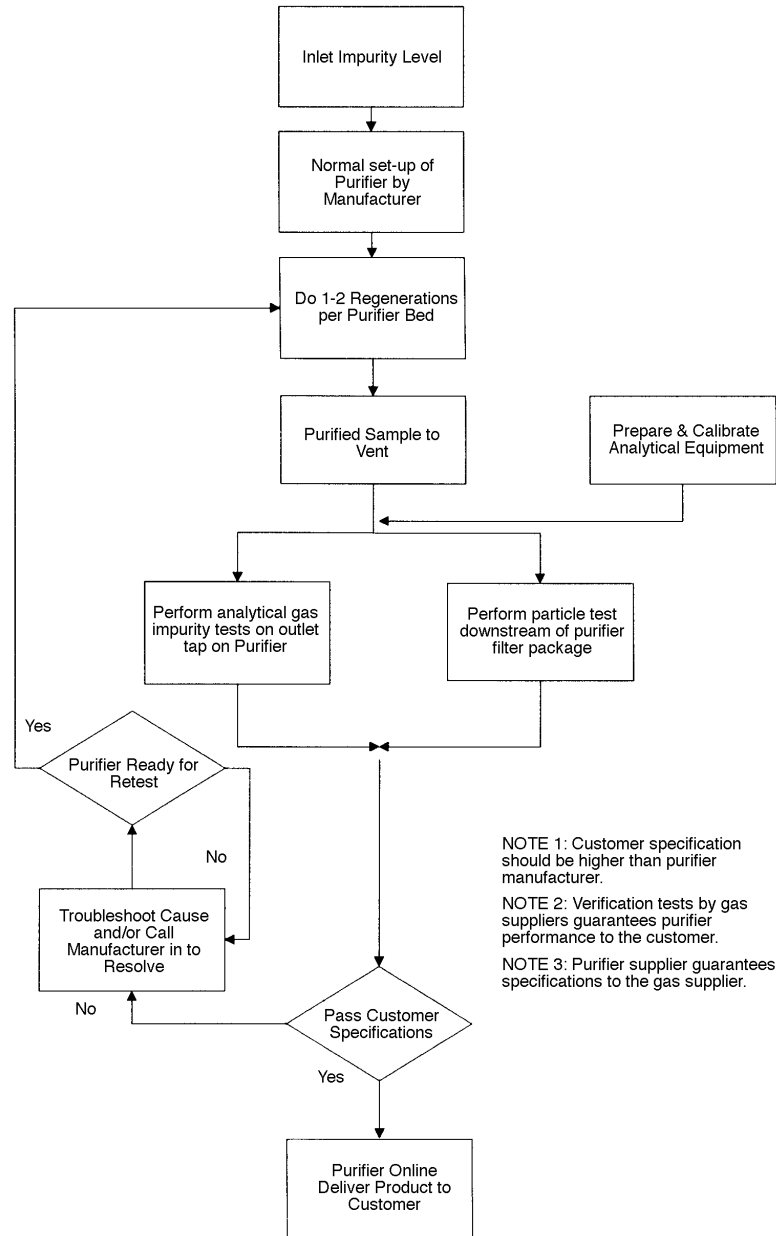


Figure 1
Overall Test Sequence

12 Preparation of Apparatus

12.1 *Sampling System* — Use appropriate clean tubing and fittings, or clean before use. Purge system prior to sampling.

12.2 *Dynamic Dilution System* — Use certified standards and dilute using equation (1) to calculate final concentration (C_F). For moisture, certified moisture permeation devices can be used.

$$(1) C_c \times D.F. = C_F$$

C_c = PPM - or PPB - certified cylinder standard concentration.

C_F = final concentration.

D.F. = dilution factor which is calculated by taking the flow (in liters) of the certified standard and dividing by total flow (in liters). Dilution factors can be multiplied in series, if diluting more than once.

Example 1: What is the final concentration for dynamically diluting 10 ml of a 100 PPB impurity into 1 liter?

$$100 \text{ PPB} \times [0.010 \text{ liter}/1.010 \text{ liter}] = 0.99 \text{ PPB}$$

12.2.1 Calibration should be done in the region specified by the purifier manufacturer. For example, if the purifier has an outlet impurity guarantee of 1 PPB of each impurity, then the test equipment should be calibrated with levels at approximately 1 PPB, not at 30 PPB and extrapolated down. Also, multipoint calibration data is preferred.

12.3 APIMS, RGD-GC, ultratrace analytical instrumentation. Particle counter.

12.3.1 Start-up and purge the instrumentation. Perform calibration. Determine if calibration is satisfactory. Proceed to sampling section.

12.4 *Data Collection System* — Check for proper signal inputs, range inputs, and sampling intervals.

12.5 *Data Reduction System* — Prepare data as print-outs and/or graphs. Include statistical analysis as required. Generate final report.

13 Calibration and Standardization

13.1 See Section 12, Preparation of Apparatus.

14 Procedure

14.1 *Trace Gas Impurity Measurement*

14.1.1 Connect sample source to analytical equipment.

14.1.2 Start data collection.

14.1.3 Stop sampling. Review preliminary data. If it is within customer specification, disconnect sample source. If it does not meet specifications, investigate

cause or refer to manufacturer literature to resolve. Once condition is corrected, repeat tests.

14.1.4 Repeat procedure for next bed or sampling point. If instrumentation is relocated, calibration check is required.

14.2 *Particle Counting*

14.2.1 Select sample location. Ideal location is a permanently installed pitot probe.

14.2.2 Select where the particle tests will be done on the pipe.

14.2.3 Use Reynold's equation to determine the required gas rate for turbulent flow to the particle counter. Use this value or higher for sampling purposes. Reynold's number greater than 2100 are suggested for turbulent flow.

14.2.4 Reduce incoming sample pressure to the particle counter by following manufacturer's recommendation or by best practice.

14.2.5 Test particles with turbulent flow through pipeline, if possible. However, do not exceed the manufacturer's maximum rated flow for the purifier.

14.2.6 Start data collection.

14.2.7 Stop sampling. Review preliminary data. If it is within customer specification, disconnect sample source. If it does not meet specifications, investigate cause or refer to manufacturer's literature to resolve. Once condition is corrected, repeat tests.

14.2.8 Repeat procedure for next bed or sampling point.

15 Calculations or Interpretation of Results

15.1 Results are interpreted by trending analysis, averaging, or steady-state analysis.

16 Reporting Results

16.1 Sample location.

16.2 Operator identification.

16.3 Test parameters and conditions (pressures, flowrates, temperatures, etc.).

16.4 Test date and duration.

16.5 Description of instrumentation.

16.6 Calibration information for analyzer(s).

16.7 Report test results by data table and/or graphs.

16.8 Comments on testing.

16.9 Conclusion.

APPENDIX 1

NOTE: This appendix was approved as an official part of SEMI F30 by full letter ballot procedure.

<i>Partial List of Analytical Equipment for Use in Oxygen Service</i>	
Impurity	Analytical Equipment
H ₂ O	Dewpoint Detection Electrolytic Piezoelectric Capacitance (aluminum oxide, silicon array) Fourier Transform Infrared (FTIR)
CH ₄ , Total Hydrocarbons, Nonmethane Hydrocarbons	Flame-Ionization Detector-Gas Chromatograph (FID-GC) Discharge Ionization Detector (DID)-GC
CO	Reduction Gas Detector (RGD)-GC Nondispersive Infrared (NDIR) DID-GC
CO ₂	Methanator on FID-GC DID-GC NDIR
H ₂	RGD-GC
Particles	Special counters required for O ₂ service: for 0.01 µ or greater, condensation nucleus counter for 0.1 µ or greater, laser counter

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SEMI F31-0698

GUIDE FOR BULK CHEMICAL DISTRIBUTION SYSTEMS

1 Purpose

1.1 This guide defines components of Bulk Chemical Distribution Systems and sets forth Basic Design Elements and optional design features common to BCD Systems.

2 Scope

2.1 This guide applies to BCD Systems used in semiconductor manufacturing facilities for supplying liquid chemicals to wafer cleaning and other manufacturing processes.

3 Limitations

3.1 This guide is not intended to be applicable to every possible use and type of chemical dispensing system but only to those systems that originate with sources of single lots of liquid materials of 55 gallons or more, and which operate in a way that splits a single liquid stream into multiple streams. This guide excludes slurry systems, small liquid chemical dispensing systems, chemical waste systems, and many other types of systems that dispense fluids.

3.2 This guide excludes construction protocols for BCD Systems, such as clean manufacturing practices, integrity of fabrication, and prequalification of materials.

3.3 This guide does not intend to cover all the important safety considerations which relate to Bulk Chemical Distribution Systems.

3.4 References to “containers” in this document are not limited to “Bulk Containers” as defined in the Code of Federal Regulations (CFR) Section 49 or Department of Transportation (DOT) Regulations.

4 Referenced Documents

NOTE: As listed or revised, all documents cited shall be the latest publications of adopted standards.

4.1 SEMI Standards

SEMI E49 — Guide for Standard Performance, Practices, and Sub-Assembly for High Purity Piping Systems and Final Assembly for Semiconductor Manufacturing Equipment

SEMI S2 — Environmental, Health, and Safety Guideline for Semiconductor Manufacturing Equipment

4.2 DOT¹

Department of Transportation

4.3 Federal Regulations²

49 CFR — Title 49 of the Code of Federal Regulations

5 Terminology

5.1 Acronyms

5.1.1 CDM — Chemical Dispensing Module

5.1.2 CDU — Chemical Dispensing Unit

5.1.3 DIW — Deionized Water

5.1.4 MDU — Modular Dispensing Unit

5.2 Definitions

5.2.1 *blending* — Combination of two or more chemicals to create a mixture which contains a desired ratio of constituents.

5.2.2 *chemical dispense system* — The module used for filtering and for dispensing chemical into the distribution piping network which may be referred to by a number of manufacturer’s specific designations: CDM, CDU, and MDU.

5.2.3 *chemical reprocessing unit* — A subsystem which purifies or recycles a chemical.

5.2.4 *chemical transfer* — The technique used to move chemical between different points in the distribution system.

5.2.4.1 *pressurization* — The use of high purity nitrogen or other appropriate gas to displace liquid through the distribution system.

5.2.4.2 *pumping* — A mechanical or pneumatically operated technique used to create hydraulic force in the system.

5.2.4.3 *hybrid system* — A combination of pressurization and pumping techniques used to transfer chemical.

5.2.5 *day tank* — A vessel that stores that amount of chemical which may be consumed within one or more days. This holding vessel usually stores prefiltered

¹ Department of Transportation, 400 Seventh Street, SW, Washington, D.C. 20590

² United States Government Printing Office, Washington, D.C. 20402

chemical until it is transferred to the manufacturing process.

5.2.6 decontamination — Cleaning up of a BCD System, or any subsystems thereof, by introducing chemical solutions into the piping systems. This procedure occurs during qualification and prior to commissioning the system.

5.2.7 dilution — Combination of a concentrated chemical and DIW to create a lower concentration of the aqueous chemical.

5.2.8 on-site gas-to-chemical generation — The contacting of a liquid and gas phase to create a stable liquid solution. The chemical is generated on-site in a form suitable for distribution through a dispense system.

5.2.9 pickling — Conditioning of the system by filling it with the actual chemical or another chemical for which the system is designed. This chemical will stay in the system for a specified period of time. The purpose of this conditioning is to leach out impurities prior to actually using any chemical in the manufacturing process.

5.2.10 polishing — The process of flowing chemical through a filter one or more times to reduce the particulate levels of the chemical.

5.2.11 primary containment — Tubing, piping, or components whose wetted surface is directly in contact with the chemical. These components are generally made from fluoropolymer materials such as Perfluoroalkoxy (PFA).

5.2.12 secondary containment — Tubing or piping which contains the primary piping or tubing. The purpose of this configuration is to control leaks and to protect against spills. Secondary containment of solvent systems generally uses stainless steel material, and acid or base systems generally use Schedule 40 Polyvinylchloride (PVC) material boxes such as VMB. This may also be used as secondary containment.

5.2.13 stabilized filtration — A design feature that provides for the continuous flow of chemical through filters to minimize shocking and pulsating of the filters.

5.2.14 surge suppression — Use of a device or in-line chamber that minimizes flow pulsations caused by a pump. This may also be referred to as pulsation dampener.

5.2.15 valve manifold box (VMB) — A chemical-resistant enclosure which houses manual and/or pneumatically actuated valves, tees, and fittings.

5.2.16 vessel — A vessel fabricated from or containing an inner lining of chemically inert and

resistant materials which is specifically designed to maintain the purity level of the chemical for a long period of time. All containers are fabricated from or at least contain an inner lining of chemically inert and resistant materials and are specifically designed to maintain the purity level of the chemical for long periods of time. The larger containers are generally designed in conformance to Department of Transportation Regulations (DOT) and may be transported by ground. These containers have fittings which are compatible to this purpose so that they may be hooked up to the BCD Systems directly. Such containers include:

5.2.16.1 drum — A container for storing chemicals, generally with a cylindrical shape and not more than 55 gallons or 200 liters in size.

5.2.16.2 tote — A container for storing chemicals, generally 110, 220, or 330 gallons in size and requiring an outer shell to provide structural support to the vessel.

5.2.16.3 ISO container — A container for storing chemicals, usually large in size, able to be transported directly, and designed in compliance with criteria from the International Standards Organization.

5.2.17 wetted surface — Any surface which comes into contact with the chemical.

6 General Requirements

6.1 Materials — Components of the BCD Systems must be appropriate to the application and conform to electrical, mechanical, and chemical requirements as defined by the physical installation environment, local and national code interpretations, process requirements, and delivery specifications.

6.2 System Installation — The Bulk Chemical Distribution System is installed according to a protocol which ensures mechanical integrity, leakproof operation, and minimal contamination to any liquids being distributed throughout the system.

6.2.1 System Installation procedures include a rinsedown protocol which may include any of the following: (1) a flushing procedure with DIW; (2) a sanitization step using hydrogen peroxide; (3) a drying step using nitrogen gas; and (4) a “pickling” process using the actual chemical, or a less concentrated form of the chemical. For solvent systems, the rinse-down chemicals introduced are generally the same as the solvent that is designated to be used in the system, whereas for acid systems, the rinse down solutions may include DIW, ozonated water, or a dilute form of hydrogen peroxide.

6.2.2 The protocol for System Installation and start-up follows a predefined sequence of events which

incorporates all of the physical activities and testing parameters and establishes check points for approvals at each stage.

6.3 Acceptance Tests — Acceptance tests are conducted on each subsystem or system produced. They are the basis for acceptance or rejection by the purchaser.

6.3.1 Acceptance testing may include performance demonstrations, demonstration of reliability criteria, and achievement of purity standards. Safety considerations are addressed elsewhere.

6.4 Qualification Testing — Qualification testing is performed on the liquid chemicals being transported through each chemical piping subsystem.

6.4.1 Qualification testing may include tests for trace metals, anions, particles, and total organic carbon.

Assay analysis is recommended for blending or dilution operations.

6.5 Chemical Specifications — Particulate BCD Systems are specified as to the level of impurities at the point of use as compared to the quality of the incoming chemical. Impurities in the chemical are generally specified in terms of particles per ml; metallic impurities are specified for each element and for total adds; and organic impurities are specified for total added.

7 Basic Design Elements

7.1 General — Each Bulk Chemical Distribution System contains certain basic components and a variety of design options to meet particular customer and facility needs. A simple example system is shown in Figure 1.

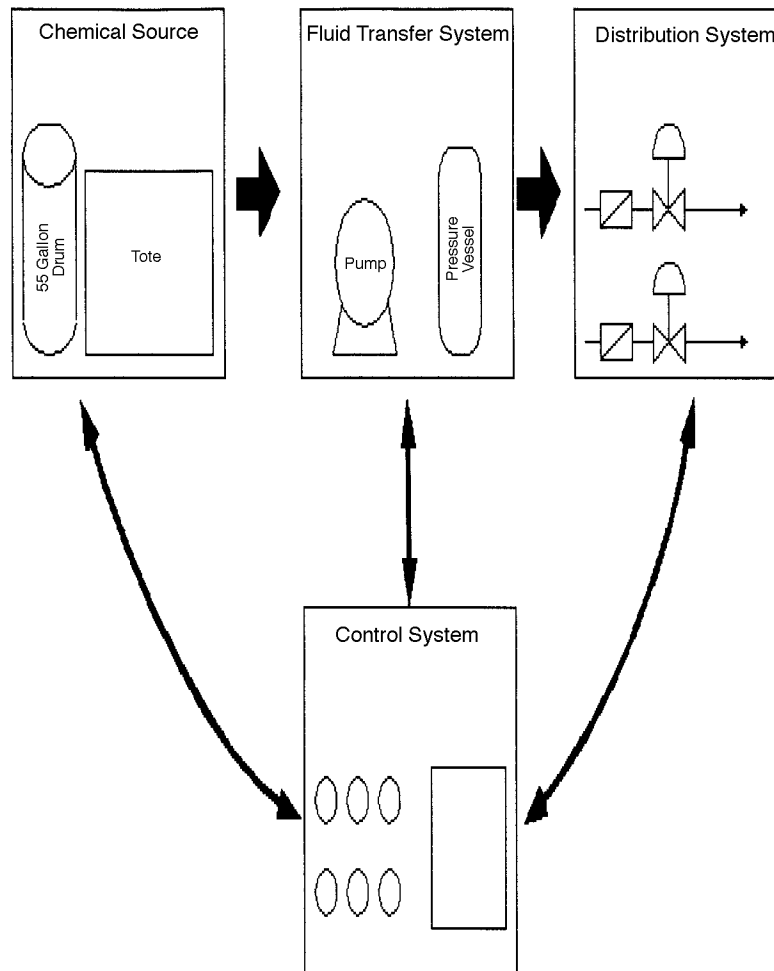


Figure 1
Schematic of Basic Bulk Chemical Distribution System

7.2 Source — The source of all fluids for incoming Bulk Chemical Distribution Systems is a container suitable for holding reactive chemicals in a size 55 gallons or greater, which may include drums, totes, vessels, or ISO containers.

7.3 Fluid Transfer — Fluids in the Bulk Chemical Distribution Systems are transported through pipes using either a pumping system, a pressure differential system, or some combination of the two types of basic systems. All fluids are transported in secondary containment piping systems for safety reasons.

7.4 Distribution — The transfer is made from the source by means of certain types of mechanisms, usually through one or more valve manifold boxes, that direct the fluid to a point of use, to subordinate levels of distribution, and/or to a day tank.

7.5 Control Systems — Manual and/or automated mechanical, pneumatic, hydraulic, optical, and electronic systems control the movement of the liquids. This includes the volume, flow rate, pressure, start-up, and shut-off.

8 Optional Design Features

8.1 Polishing — BCD Systems may be designed with the possibility of chemical recirculation (1) from the incoming source and back, to polish the incoming chemical; (2) from the day tank and back for further polishing or timed recirculation if activity is limited; and (3) fabwide in continuous recirculation.

8.2 Filtration — Chemicals in the system may be recirculated through filters placed on-line in the system. The relevant design issues include types of filters, efficiency, useful lifetime, qualification, filter bleed, removal, and replacement.

8.3 Day Tank — The vessel that generally stores a limited amount of chemical volume generally equivalent to that used in about one day of operation. This holding vessel usually stores prefiltered chemical before it is transferred to the manufacturing process.

8.4 Blending — Certain chemicals in distribution are blended together for use in the semiconductor manufacturing process. For example, Buffered Oxide Etchant is a blend of Hydrofluoric Acid and Ammonium Fluoride and may be sourced as two separate chemicals and blended together in the correct concentrations within the BCD Systems.

8.5 Dilution — Certain chemicals used in the semiconductor manufacturing process require various concentrations, depending on the process used at a particular point in the system. For example, 49% Hydrofluoric Acid is brought in as the source chemical

and then mixed with DIW in a blend unit to achieve various concentrations of 10:1, 100:1, etc., depending on the requirements of the manufacturing process.

8.6 Automated Control Systems — Electronic systems may be programmed to control system parameters such as volume, pressure, fill rate, flow, drain times, maintenance diagnostics, purge cycles, recirculation, and bypass.

8.7 Sampling Ports — Sampling ports may be incorporated into the system to facilitate chemical sampling for qualification and monitoring programs.

8.8 On-Site Chemical Generation — Liquid chemicals, such as Ammonium Hydroxide, may be generated on-site at the required concentration from a clean Ammonia gas source and DIW to the required concentration. Generally, this activity is done in a location apart from the dispense room. The resulting chemical is used as a source for the distribution system.

8.9 Reprocessing — Integrated with the general chemical distribution system may be a chemical reprocessing unit which purifies or recycles a chemical such as sulfuric acid, isopropanol, or hydrofluoric acid. Such a system enables the chemical to reenter the system as a clean source of material for manufacturing processing.

8.10 Stabilized Filtration — Some systems include a design feature that provides for the continuous flow of chemical over filters to minimize shocking and pulsating of the filters.

8.11 Surge Suppression — Some systems include a device or in-line chamber that minimizes hydraulic forces caused by a pump. This option may also be referred to as pulsation dampener.

9 Monitoring and Maintenance of Systems

9.1 General Considerations — Bulk Chemical Distribution Systems may require a significant level of maintenance and monitoring to determine that specifications and performance are initially and continually met.

9.2 Functional Management System — The entire dispense system can be monitored by means of PC/PLC/ or any hybrid system, recording events as they occur and alerting operators as necessary to take corrective action. Monitoring systems may include a variety of operational parameters:

9.2.1 on-line real-time monitoring of all chemical dispense units

9.2.2 status of components and subsystems

9.2.3 events logging

- 9.2.4 data management
- 9.2.5 reliability parameters, including lead detection
- 9.2.6 chemical usage
- 9.2.7 display of dispense systems
- 9.2.8 communication gateway to the factory alarm system
- 9.2.9 on-line documentation
- 9.2.10 predictive maintenance planning
- 9.2.11 chemical interruptions to the fab by location, indicating location of fault.

9.3 *Maintenance* — Maintenance of the BCD Systems includes routine and preventative maintenance of pumps, filters, valves, and other components.

9.4 *Monitoring Programs* — Each BCD System should be monitored to determine if liquid chemicals delivered to the tools for use in the manufacturing process continually meet specifications and are within initial and ongoing established process control limits.

9.4.1 Monitoring programs may include periodic testing of the liquid chemicals to determine levels of particles, trace metals assay, and other parameters of interest. Certain tests may be done continuously on-line (e.g., particles), and other analytical tests are performed periodically from samples taken at various points in the system.

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SEMI F32-0998

TEST METHOD FOR DETERMINATION OF FLOW COEFFICIENT FOR HIGH PURITY SHUTOFF VALVES

1 Purpose

1.1 This test method describes how to determine two criteria used in selecting valves of appropriate size for gases and liquids.

1.2 Methods and equations are specified and/or referenced to assist in accurate calculation of pressure drops across valves tested by this method.

2 Scope

2.1 This method establishes the testing criteria for determination of two coefficients specified in ANSI/ISA-S75.02:

- Valve flow coefficient (C_v)
- Critical pressure drop ratio factor (x_T)

2.2 This method is to be used with ANSI/ISA-S75.02. This method applies to manual and actuated valves for use in both gas and liquid distribution systems used in semiconductor manufacturing facilities. It is a test method, where existing test methods are referenced and limitations are imposed on test conditions. Specific equations for calculating flow coefficients, choked flow parameters, and pressure drops are referenced to the appropriate ISA section.

3 Limitations

3.1 This method will limit the use and interpretation of ANSI/ISA-S75.02 for use by manufacturers and users of valves designed for the semiconductor industry.

3.2 This method is not intended to be used to determine flow coefficients for valves used in vacuum service.

4 Referenced Documents

NOTE: As listed or revised, all documents cited shall be the latest publications of adopted standards.

4.1 ANSI Documents¹

ANSI/API 2530 — Part 2: Natural Gas Fluids Measurement - Concentric, Square-Edged Orifice Met

ANSI/ISA-S-75.01 — Flow Equations for Sizing Control Valves

ANSI/ISA-S75.02 — Control Valve Capacity Test Procedure

5 Terminology

5.1 Acronyms

5.1.1 ΔP — Pressure drop across valve, kPa (psi).

5.1.2 F_k — Ratio of specific heats factor, where:

$$F_k = \frac{k}{1.40}$$

5.1.3 F_L — Liquid pressure recovery factor

$$F_L = \frac{Q_{\max}}{0.0865 * C_v * \sqrt{\frac{P_1 - 0.96 * P_v}{S_f}}}$$

$$F_L = \frac{Q_{\max \text{ gpm}}}{1.00 * C_v * \sqrt{\frac{P_1 \text{ psia} - 0.96 * P_v \text{ psia}}{S_f}}}$$

5.1.4 gpm — Gallons per minute

5.1.5 k — Specific heat ratio

5.1.6 P_1 — Absolute pressure at upstream pressure tap, kPa (psi).

5.1.7 P_2 — Absolute pressure at downstream pressure tap, kPa (psi).

5.1.8 P_v — Absolute vapor pressure of liquid at inlet temperature, kPa (psi).

5.1.9 $psia$ — Pounds per square inch absolute

5.1.10 $psid$ — Pounds per square inch differential

5.1.11 Q — Volumetric flow rate

5.1.12 Q_{\max} — Maximum flow rate (choked flow conditions) at a given upstream condition.

5.1.13 $scfh$ — Standard cubic feet per hour

5.1.14 S_f — Specific gravity of a liquid relative to water.

5.1.15 S_g — Specific gravity of a gas relative to air.

5.1.16 T — Absolute temperature of test gas or liquid, °K (°R).

¹ American National Standards Institute, 11 West 42nd St., New York, NY 10036, Telephone: 212.642.4900, Fax: 212.398.0023

5.1.17 x — Ratio of pressure drop to absolute inlet pressure, dimensionless, where:

$$x = \frac{\Delta P_{kPa}}{P_{1kPa}} \quad | \quad x = \frac{\Delta P_{psid}}{P_{1psia}} \quad |$$

5.1.18 x_T — Ratio of pressure drop to absolute inlet pressure ($\Delta p/p_1$) at choked flow condition, dimensionless.

5.1.19 Y — Expansion factor for compressible fluids, where:

$$Y = 1 - \frac{x}{3 \times F_k \times x_T}$$

5.2 Definitions

5.2.1 *flow coefficient* C_v — A numeric constant used to characterize the flow capacity of a valve.

5.2.2 *vapor pressure condensation point* — Pressure at which fluid phase changes from liquid to gas, for a given upstream condition.

5.2.3 *vena contracta* — Point in a duct where the diameter of the fluid stream is smaller than the diameter of the duct.

6 Test Fluids

6.1 *Incompressible (Liquid) Fluid* — Water is the standard liquid test fluid.

6.2 *Compressible (Gaseous) Fluid* — Nitrogen is the standard gaseous test fluid. When using Nitrogen, care should be taken to assure that the fluid does not approach the vapor pressure condensation point at the vena contracta.

7 Test Setup

7.1 Test Valve

7.1.1 The test valve can be any high purity valve, or a combination of valve with tube connections, fittings connection, or expanders which are normally attached as part of the valve assembly as purchased. It is important to note that the definition of the “test valve” is inclusive of all connections and fittings, as supplied by the manufacturer. This specifically differs from the ISA procedure, whereby a method is provided to differentiate the pressure drop contribution of the attached fittings. It is recognized that flow coefficients may vary slightly depending upon the end connection used. Examples of typical test valves are shown in Figure 1.

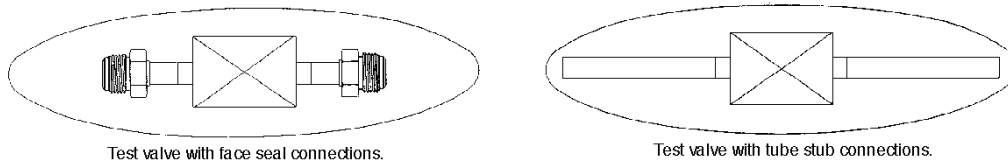


Figure 1
Test Valve with Various Connections

7.2 Test Section Requirements

7.2.1 Fixturing of the test valve shall be made in accordance with ANSI/ISA-S75.02, Table 1, where the test valve is the complete valve assembly including connections, as described above. For reference, ANSI/ISA-S75.02 test setup is shown in Figure 2.

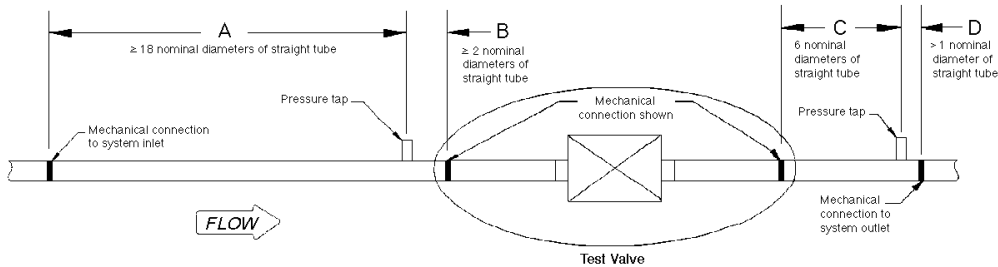


Figure 2
Fixturing Requirements, Standard Test Section

7.2.2 *Tube Connections* — Connections to the test valve are to be the same as would normally be performed by the end user as installed. Tube connections are to be full penetration welded by automatic orbital head, with purge gas.

7.2.3 *Face Seal Connections* — Face seal connectors supplied with the test valve may be either male or female. An appropriate mating connector shall be welded into the inlet and outlet tubing using full penetration orbital head welds, and connected to the valve with standard seals.

7.2.4 *Compression Fitting Connections* — Valves supplied with tube stubs may be connected with compression fittings.

8 Test Procedure

8.1 *Setup* — The test setup shall be as shown in ANSI/ISA-S75.02, Section 3.1. For reference, see Figure 3. The flowmeter may be upstream or downstream of the test valve, and can also be placed downstream of the throttle valve when necessary to vent to atmosphere.

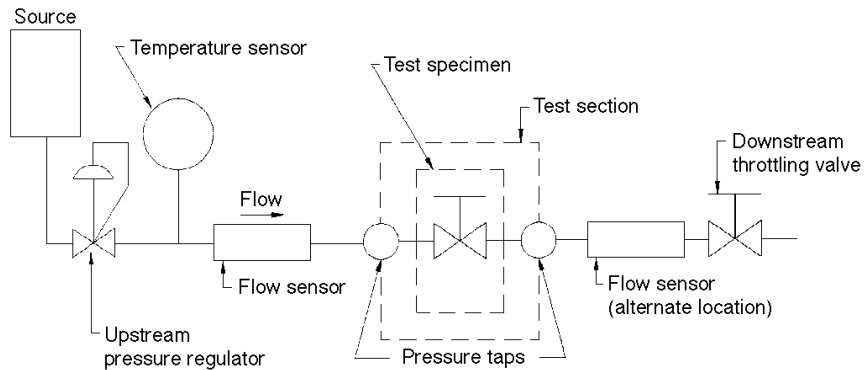


Figure 3
Test Setup

8.1.1 *Pressure Taps* — Pressure taps should be welded into the system per ANSI/ISA-S75.02.

8.1.2 *Pressure Measurement* — Pressure measurement devices can be analog gauge, electronic transducer, or both. They should be calibrated to maintain the minimum accuracy necessary for the governing flow equations.

8.1.3 *Throttle Valve(s)* — A throttle valve shall be positioned downstream. There is no valve style restriction, however they should be sized sufficiently for the required flowrate.

8.1.4 *Flow Measurement* — Depending upon the test media, several types of flow measurement devices may be used. These devices may include: turbine meter, orifice plate, bell prover, mass flow meter, and timed volumetric capture. Flow measurement devices should be calibrated to maintain the minimum accuracy necessary for the governing flow equations. All flow measurements should be normalized to standard conditions per ANSI 2530. Install and calibrate per manufacturer's recommended procedure.

8.1.5 *Temperature Measurement* — Temperature measurement devices should be calibrated to maintain the minimum accuracy necessary for the governing flow equations.

8.2 Incompressible Fluid

8.2.1 Procedure

8.2.1.1 The test procedure is specified in ANSI/ISA-S75.02, Section 5.1.

8.2.2 Test Limitations

8.2.2.1 It is critical that the absolute upstream pressure meets the criteria specified in ANSI/ISA-S75.02, paragraph 5.1.3, as referenced below:

$$P_{1_{kPa}} = \frac{2 \times \Delta P_{kPa}}{F_L^2} \mid P_{1_{psia}} = \frac{2 \times \Delta P_{psid}}{F_L^2} \mid$$

Equation 1

Absolute Upstream Pressure

8.2.2.2 The liquid pressure recovery factor, F_L is calculated from the maximum attainable flowrate, Q_{max} . If Q_{max} cannot be experimentally determined, the value of F_L can be estimated. See the referenced section for details.

8.3 Compressible Fluid

8.3.1 Procedure

8.3.1.1 The test procedure is specified in ANSI/ISA-S75.02, paragraph 7.1.

8.3.2 Test Limitations

8.3.2.1 If possible, at least 3 data points should be taken at flowrates where the fluid approaches incompressible behavior. To attain these conditions, the pressure drop ratio, x , should be less than or equal to 0.02. Additional data points should be taken at increasing flowrates up to the maximum possible for the system.

9 Coefficients Calculation

9.1 Flow Coefficient, C_v

9.1.1 The flow coefficient, C_v , can be calculated using both incompressible and compressible fluids. For incompressible fluids, ISA provides a relatively simple method for determining C_v . For compressible fluids, the ISA test method for calculating C_v requires the determination of the choked flow pressure drop ratio, x_T , and ultimately the expansion factor, Y , which characterizes the valve geometry and fluid properties at sonic velocities in the vena contracta. It has been determined through considerable testing that the expansion factor is critical in calculating actual pressure drops across a valve when flow rates cause the fluid density to change due to pressure and velocity changes.

9.2 Pressure Drop Ratio Factor, x_T

9.2.1 The ISA test method requires the experimental determination of the flow coefficient C_v and a second coefficient x_T , the pressure drop ratio factor.

9.2.2 x_T is equal to the critical pressure ratio of the ISA flow equation, and is close but not necessarily identical to the experimentally observed pressure ratio at choked flow. x_T is chosen to correlate the experimentally observed choked flow. The ISA equation will then predict choking at a pressure drop ratio close to, but not identical with, the experimentally observed value.

9.2.3 There are two methods to determine the pressure drop ratio factor. The procedure of ANSI/ISA-S75.02, Section 7.2 requires that C_v be determined at values of x less than 0.02, and that choking actually be achieved to determine x_T . The alternate procedure of paragraph 7.3 does not actually require choked flow and uses all the data points to determine C_v , not just a few points at a small value of x .

9.2.4 In practice, valves used in the semiconductor manufacturing facility, when connected as described above, may not be able to achieve choked flow across the pressure taps provided by this procedure. This will happen when choked flow occurs first in some other part of the test setup or connections. However, the alternative test procedure of ANSI/ISA-S75.02, Section 7.3 will give the correct values of C_v and x_T in this case.

9.2.5 The ISA alternative method determines the pressure drop ratio factor by measuring flowrate vs.

pressure ratio, and analyzes the data to compute the choked flow pressure drop ratio, x_T . This method has been found, through exhaustive testing, to be a very accurate and reliable method to determine the expansion factor, without having to achieve choked flow in the test valve itself.

10 Pressure Drop Calculation

10.1 The determination of both flow coefficient and expansion factor are critical in calculating the pressure drop across a valve flowing compressible fluid. Use of the flow coefficient C_v only, will yield a pressure drop which is lower than the actual value.

10.1.1 Incompressible Flow

10.1.1.1 To calculate pressure drop across a valve flowing an incompressible media, only the C_v coefficient must be provided by the valve manufacturer. The following equation has been derived from ANSI/ISA-S75.01, Section 4.1, and should be used for pressure drop computations:

$$\Delta P_{kPa} = 0.0865 * \left| \frac{Q_{m^3/hr}}{C_v} \right|^2 \times S_f$$

$$\left(\Delta P_{psid} = \left(\frac{Q_{gpm}}{C_v} \right)^2 \times S_f \right)$$

Equation 2
Pressure Drop for Incompressible Flow

10.1.2 Compressible Flow

10.1.2.1 To calculate pressure drop across a valve flowing a compressible media, both C_v and x_T coefficients may be used to determine more accurately the pressure drop. Contact the valve manufacturer for values of C_v and x_T coefficients. Equation 3 has been derived from ANSI/ISA-S75.01, Section 6.1, and should be used for pressure drop computations. Equation 4 is a close approximation of Equation 3 that computes pressure drop directly.

$$\Delta P_{kPa} = \left| \frac{Q_{m^3/hr}}{4.17 \times C_v \times Y \times P_{1kPa}} \right|^2 \times S_g \times T_o \times P_{1kPa}$$

$$\left(\Delta P_{psid} = \left(\frac{Q_{scfh}}{1360 \times C_v \times Y \times P_{1psia}} \right)^2 \times S_g \times T_o \times P_{1psia} \right)$$

Equation 3
Pressure Drop for Compressible Flow – ISA
Iterative Solution

$$\Delta P = \frac{P_{1kPa} \times x_T}{1.125} \left[1 - \sqrt{1 - \frac{1.125}{x_T} \left| \frac{S_g \times Q_{m^3/hr}}{3905.6 \times C_v \times P_{1kPa}} \sqrt{\frac{T_o}{S_g}} \right|^2} \right]$$

$$\left(\Delta P = \frac{P_{1psia} \times x_T}{1.125} \left[1 - \sqrt{1 - \frac{1.125}{x_T} \left(\frac{S_g \times Q_{scfh}}{16.04 \times C_v \times P_{1psia}} \sqrt{\frac{T_o}{S_g}} \right)^2} \right] \right)$$

Equation 4
Pressure Drop for Compressible Flow – Non-Iterative Solution

NOTE: The numerical value of x used in these equations must not exceed the choking limit (x_T) regardless of the actual value of x .

11 Related Documents

11.1 ISO Document²

ISO 6358 — Pneumatic fluid power — Components using compressible fluids — Determination of flow-rate characteristics

11.2 SAE Documents³

ARP 24B — Determination of Hydraulic Pressure Drop

ARP 868 — Pressure Drop Test for Fuel System Components

² International Organization for Standardization, Casa Posatale 56, CH-1211, Geneva 20, Switzerland

³ Society of Automotive Engineers, World Headquarters, 400 Commonwealth Dr., Warrendale, PA 15096-0001, Telephone: 724.776.4841, Fax: 724.776.5760



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SEMI F33-0998

METHOD FOR CALIBRATION OF ATMOSPHERIC PRESSURE IONIZATION MASS SPECTROMETER (APIMS)

1 Purpose

1.1 This test method may provide guidelines for the calibration of the APIMS for measurement of impurities in nitrogen, argon, helium and hydrogen. APIMS is currently the technique of choice for measurements of low level impurities in gas distribution systems and components because it is essentially the only commercially available method capable of ppt impurity analysis and it has a superior response time. This method may provide guidelines for application of other techniques with similar detection limits and response time to APIMS which are not commercially available at this time.

2 Scope

2.1 This method applies to the analyte calibration of the APIMS for a target impurity range of 100 ppt to as high as 100 ppb impurity range. The actual calibration range should bracket the impurity measurement range of interest, dependent upon the measurement to be conducted. Anything else is outside the range of the calibration.

3 Limitations

3.1 The actual range of calibration will depend upon the type of APIMS used. Counting detection electronics will saturate in the 200,000 CPS to 1,000,000 CPS range, depending upon the make and model. Counting detection will have to use different ions in different concentration regions, depending upon the impurity. The actual calibration procedure will be different for APIMS using analog detection versus counting detection. Interference between impurities can cause limitation in measurements in the presence of multiple impurity species.

4 Referenced Documents

4.1 SEMI Standards

SEMI C9.1 — Guide for Analysis of Uncertainties in Gravimetrically Prepared Gas Mixtures

SEMI C15 — Test Method for ppm and ppb Humidity Standards

5 Terminology

5.1 Acronyms

5.1.1 *APIMS* — Atmospheric Pressure Ionization Mass Spectrometer

5.1.2 *CPS* — Counts per second

5.1.3 *m/z* — *m* in atomic mass units and *z* in elementary charge units

5.1.4 *NIST* — National Institute of Standards and Technology

5.1.5 *ppb* — Molar parts per billion (nmole/mole). The same as ppbv.

5.1.6 *ppm* — Molar parts per million (μmole/mole). The same as ppmv.

5.1.7 *ppt* — Molar parts per trillion (pimole/mole). The same as pptv.

5.1.8 *R*² — The statistic described by the ratio of the sum of squares of the regression divided by the total sum of the squares.

5.2 Definitions

5.2.1 *zero gas* — Nitrogen, argon, helium or hydrogen with an estimated level an order of magnitude, or more, lower than the lowest calibration point for each impurity of interest.

6 Summary of Method

6.1 The calibration of the APIMS is conducted by adding known concentrations of impurities to a zero gas and measuring the corresponding ion intensities. A calibration response factor can be determined by regression analysis.

7 Interferences

7.1 It is essential to confirm that the mass chosen is indeed representative of the species of interest and that other commonly present impurities do not contribute at the same mass. For example: the intensity of the peak at *m/z* = 29 correlates with the concentration of H₂ in N₂, because of the N₂H⁺ formation. However, spurious signal at this peak can arise in two different ways:

(i) Contributions of other ions of the same mass, such as N¹⁴N¹⁵⁺ or C₂H₅⁺.

(ii) Contributions of the same ion from different parent species, i.e., N₂H⁺ due to recombination of matrix gas nitrogen with fragments of H₂O, CH₄, etc. Before using *m/z* = 29 as a measure of H₂ in N₂, interference such as these must be understood. This also may apply to other ions.

7.2 High impurity levels can cause clustering with, or quenching of, the other impurity ions and affect the sensitivity of the APIMS for those impurities. This is especially true for impurity levels of 10 ppb and above.

8 Apparatus

8.1 *APIMS* — The APIMS used for impurity detection can be of any type.

8.2 *Impurity Standard* — Any device (either standard cylinder or permeation tube based or of any other type) that can reliably deliver impurity concentrations usually in the range of 100 ppb to 10 ppm with an accuracy of $\pm 5\%$. The standards should be traceable to an applicable national standard, such as NIST.

8.3 *Dynamic Dilution System* — A dilution system that can dilute the output gas of the impurity standard with a zero gas to produce a gas with impurity concentrations in the range of interest.

9 Reagents and Materials

9.1 Zero gas

9.2 Impurity standard

10 Safety Precautions

10.1 This method applies to calibration and measurement of impurities in nitrogen, argon, helium and hydrogen. This method does not address the additional safety precautions for APIMS calibration and impurity measurement in hydrogen gas.

11 Test Specimen

11.1 Not applicable.

12 Preparation of Apparatus

12.1 The impurity standard and zero gas should be connected to the dynamic dilution system, which is in turn connected to the inlet of the APIMS. The entire dilution system should be purged until all impurities have stabilized at a constant dilution system flow, for all components in the dilution system. The relative standard deviation for each analyte should be less than 10% for the past 12 hours before the calibration. Constant pressure and flow should be maintained to the APIMS ionization region at all time.

13 Calibration and Standardization

13.1 *Definition of an Impurity Standard* — An impurity “standard” is defined to be any device capable of delivering a flow of known impurity level gas at a controlled pressure. (See Section 8.2.)

13.2 *Examples of Standards* — Some examples of impurity standards and the principles upon which they are based are as follows:

13.2.1 *Cylinder Standards* — The “standard” will include a regulator specified for use with the cylinder. The impurity concentration can be calculated based on that added to the cylinder in preparation. A minimum use pressure must be specified.

13.2.2 Permeation and effusion tube standards. These must include purification means and some means of regulating the outlet pressure. The impurity delivery rate can be calculated based on the weight loss of the tube as a function of time.

13.2.3 Moisture standard methods based on saturation of gas with water vapor at a fixed temperature and pressure. The moisture concentration in the gas may then be calculated from a knowledge of the saturation vapor pressure of water over a plane of the pure phase of ice at the saturation temperature and of the interaction virial coefficients of the gas-vapor mixture. The two-pressure and two temperature methods are refinements of this approach requiring additional chambers whose temperature and pressure must be known.

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

13.3.1 The zero gas should have an impurity level at least an order of magnitude below the stated level of the analysis (to be verified using the same flow path as during the subsequent analysis, and at the lowest flow rate actually used in the calibration).

13.3.2 The absolute accuracy of the dilution system components are verified by comparison with a reliable flow standard. The frequency of verification must take into account the transport of the instrument and other changes that will affect the dilution system.

13.3.3 The linearity of the dilution system can be demonstrated over the entire range of operation. This last criterion is particularly important whenever some portion of the combined flow is discarded, as mixing problems can easily arise at large dilution factors.

13.4 *Mass Calibration* — The mass scale of the mass spectrometer should be calibrated according to the manufacturer’s instructions over the mass range of interest.

13.4.1 The validity of the mass selected for each impurity must be confirmed (i) by demonstrating that the intensity at the mass increases monotonically with added concentration of the impurity species of interest and (ii) that other usually present impurity species do not make significant contributions to the intensity at that mass. Tables 1–4 list the typical masses for various species in argon, nitrogen, hydrogen and helium.

Table 1 Common Masses in Argon Bulk Gas

<i>m/z</i>	<i>Ions</i>	<i>Source</i>
14	N ⁺	Analyte
15	CH ₃ ⁺	Analyte
16	CH ₄ ⁺	Analyte
17	OH ⁺	Analyte
18	H ₂ O ⁺	Analyte
19	H ₃ O ⁺	Analyte
20	Ne ⁺	Bulk Gas Impurity
28	CO ⁺	Analyte
32	O ₂ ⁺	Analyte
36	Ar ⁺	Bulk Gas Isotope
37	(H ₂ O) ₂ H ⁺	Analyte
38	Ar ⁺	Bulk Gas Isotope
40	Ar ⁺	Bulk Gas
41	ArH ⁺	Analyte
44	CO ₂ ⁺	Analyte
68	ArCO ⁺	Analyte
68	ArN ₂ ⁺	Analyte
76	Ar ₂ ⁺	Bulk Gas Isotope
78	Ar ₂ ⁺	Bulk Gas Isotope
80	Ar ₂ ⁺	Bulk Gas

NOTE: In APIMS, the same ion may be formed even though different parent molecules are introduced into the ionization region. Thus, it is necessary to be sure that the chosen *m/z* represents the ion of interest and that the ion of interest originates from the molecule of interest.

Table 2 Common Masses in Nitrogen Bulk Gas

<i>m/z</i>	<i>Ions</i>	<i>Source</i>
12	C ⁺	Analyte
14	N ⁺	Bulk Gas
15	CH ₃ ⁺	Analyte
16	CH ₄ ⁺	Analyte
17	OH ⁺	Analyte
18	H ₂ O ⁺	Analyte
19	H ₃ O ⁺	Analyte
28	N ₂ ⁺	Bulk Gas
28	CO ⁺	Analyte
29	N ₂ H ⁺	Analyte
29	COH ⁺	Analyte
29	C ₂ H ₅ ⁺	Analyte
29	N ₂ ⁺	Bulk Gas Isotope
32	O ₂ ⁺	Analyte
37	(H ₂ O) ₂ H ⁺	Analyte
42	N ₃ ⁺	Bulk Gas
43	N ₃ H ⁺	Analyte
44	CO ₂ ⁺	Analyte
44	N ₂ O ⁺	Analyte
46	N ₂ H ₂ O ⁺	Analyte
55	(H ₂ O) ₃ H ⁺	Analyte
56	N ₄ ⁺	Bulk Gas
70	N ₅ ⁺	Bulk Gas

NOTE: In APIMS, the same ion may be formed even though different parent molecules are introduced into the ionization region. Thus, it is necessary to be sure that the chosen *m/z* represents the ion of interest and that the ion of interest originates from the molecule of interest.

Table 3 Common Masses in Hydrogen Bulk Gas

<i>m/z</i>	<i>Ions</i>	<i>Source</i>
2	H ₂ ⁺	Bulk Gas
17	CH ₅ ⁺	Analyte
18	H ₂ O ⁺	Analyte
19	H ₃ O ⁺	Analyte
29	N ₂ H ⁺	Analyte
29	COH ⁺	Analyte
33	O ₂ H ⁺	Analyte
34	O ₂ H ₂ ⁺	Analyte
35	O ₂ H ₃ ⁺	Analyte
37	(H ₂ O) ₂ H ⁺	Analyte
45	CO ₂ H ⁺	Analyte

NOTE: In APIMS, the same ion may be formed even though different parent molecules are introduced into the ionization region. Thus, it is necessary to be sure that the chosen *m/z* represents the ion of interest and that the ion of interest originates from the molecule of interest.

Table 4 Common Masses in Helium Bulk Gas

<i>m/z</i>	<i>Ions</i>	<i>Source</i>
4	He ⁺	Bulk Gas
5	HeH ⁺	Analyte
8	He ₂ ⁺	Bulk Gas
9	He ₂ H ⁺	Analyte
12	He ₃ ⁺	Bulk Gas
12	C ⁺	Analyte
14	N ⁺	Analyte
15	CH ₃ ⁺	Analyte
16	He ₄ ⁺	Bulk Gas
16	CH ₄ ⁺	Analyte
18	H ₂ O ⁺	Analyte
20	Ne ⁺	Bulk Gas Isotope
22	HeH ₂ O ⁺	Analyte
24	NeHe ⁺	Bulk Gas Impurity
26	He ₂ H ₂ O ⁺	Analyte
28	N ₂ ⁺	Analyte
28	CO ⁺	Analyte
28	He ₇ ⁺	Bulk Gas
32	O ₂ ⁺	Analyte
40	Ar ⁺	Bulk Gas Impurity
44	CO ₂ ⁺	Analyte
44	ArHe ⁺	Bulk Gas Impurity

NOTE: In APIMS, the same ion may be formed even though different parent molecules are introduced into the ionization region. Thus, it is necessary to be sure that the chosen *m/z* represents the ion of interest and that the ion of interest originates from the molecule of interest.

14 Procedure

14.1 Zero gas should be passed through the dilution system and the APIMS. Based on prior calibrations, the system should be below 100 ppt, or at least an order of magnitude lower than the lowest calibration point, for the impurities to be calibrated.

14.2 Prior to beginning the calibration, record all relevant parameters, such as discharge voltage, lens voltages, ionization chamber pressure and/or flow, gas type, etc.

14.3 The user should use the zero gas only, plus a minimum of 5 challenge concentrations, with a minimum of 5 data points, with the blended data points spanning the range of interest of impurity measurement, excluding the zero point, while maintaining constant flow to the APIMS. Allow each challenge concentration to stabilize for at least 15 minutes, and the signal variation should be less than 3% before taking data points at that concentration. Record the relevant ion intensities as a function of time, for each stabilized concentration.

14.4 Once the linearity of the calibration has been established, a single point calibration check is permitted, as long as the accuracy of the dilution system is confirmed.

15 Calculations or Interpretation of Results

15.1 Perform a regression analysis for the individual ion intensities against the impurity concentration. Be sure to use only ion intensities below the saturation region of your APIMS ion counting electronics, if using counting detection. (Ask the manufacturer for the saturation region of the units counting electronics.) The R^2 from the fit of all the regression coefficients should be 0.98 or above. If this is not the case, the calibration will have to be repeated as described in the previous section.

16 Reporting Results

16.1 As a minimum, the calibration will include the following information for each impurity:

$$y = c_0 + c_1 * x \quad (1)$$

where,

y is the ion intensity, c_0 is ion intensity background, c_1 is the linear response factor, and x is the impurity level.

16.2 As an option, a quadratic regression may be reported for the calibration, and will include the following information for each impurity:

$$y = c_0 + c_1 * x + c_2 * x^2 \quad (2)$$

where,

y is the ion intensity, c_0 is ion intensity background, c_1 is the first order response factor, c_2 is the second order response factor and x is the impurity level.

The c_0/c_1 ratio or negative x - intercept is useful to define the background concentration of each particular ion. All regression coefficients should be reported.

17 Precision and Accuracy

17.1 This method does not explicitly consider the estimation of the accuracy of the impurity standard in detail, because such a calculation can be made by applying procedures described elsewhere. The precision and accuracy required in validation will vary with the proposed application and may be left to the discretion

of the user of the method. If an accuracy statement is given, then a propagation of error calculation must be provided to estimate the accuracy of the validation.

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The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

SEMI F34-0998

GUIDE FOR LIQUID CHEMICAL PIPE LABELING

1 Purpose

1.1 This document establishes a recommended scheme for labeling liquid process chemical piping systems used in semiconductor manufacturing facilities and equipment.

1.2 consistent labeling system facilitates rapid identification of piping contents and avoids confusion within the industry by eliminating the need for each company or locality to develop their own guides.

2 Scope

2.1 This guide applies to components typically found in a liquid process chemical piping system for semiconductor manufacturing processes. The following list of included components is not exhaustive:

- Piping
- Valves
- Filters
- Regulators
- Tubing

3 Limitations

3.1 This guide does not replace or supersede any labeling or identification requirements established by local, state, or national authorities. Local laws and regulations should be reviewed to ensure compliance.

3.2 This guide does not apply to containers regulated by the United States Department of Transportation (DOT), International Air Transport Association (IATA) or other regulatory bodies.

3.3 This guide was prepared to address piping systems conveying liquid process chemicals located within semiconductor facilities. See Sections 6.6 and 6.11 for application inside equipment.

4 Referenced Documents

NOTE: All documents cited should be the latest published versions.

4.1 ANSI Document¹

ANSI/ASME A13.1 — Scheme for the Identification of Piping Systems

5 Terminology

5.1 *liquid chemical distribution system* — The collection of components and subsystems used to control and deliver liquid process chemicals from a source location to a point of use in a semiconductor manufacturing facility.

6 Recommendations

6.1 Piping system labels and identification, should be designed, and implemented in accordance with the following recommendations.

6.2 The following color schemes should be used to identify all liquid process chemical piping systems. When a chemical has characteristics of more than one category, the color scheme should be determined by the most significant health or safety hazard of the chemical.

6.3 Background colors may be applied to the piping by using pigments, paints, or other means acceptable to the owner; or may be applied as the background color for labels or tags used to identify the contents of the piping system.

6.4 Primary label information consists of the name of the material contained in the piping system; spelled out in the predominant language of the country or region where the piping system is used. Additional languages may be used to supplement the primary language.

¹ American National Standards Institute, 11 West 42nd Street, 13th floor, New York, NY 10036, USA, Telephone: +1.212.642.4900, Telefax: +1.212.398.0023, E-mail: info@ansi.org, WWW: <http://www.ansi.org/>

Table 1 Piping and Label Color Codes

<i>Chemical Category</i>	<i>Background Color</i>	<i>Text Color</i>
Highly Toxic, Poisonous	Black	White
Flammable, Combustible, and Pyrophoric	Red	White
Acidic	Orange	Black
Basic (Caustic)	Magenta	White
Oxidizer	Yellow	Black
Non-Toxic, Non-Flammable, Low to Moderate Physical Hazard	Green	White

6.5 Additional information such as the chemical category, chemical formula, acronyms, hazard ratings, or other information may be added, provided that it does not interfere with the primary information required. A separate label may be used for the additional information.

6.6 The color field label length and lettering sizes shown in Table 2 should be used for all diameters of pipe and tubing. For pipe diameters smaller than 12.7 mm (0.5 inch), the label should be large enough to wrap at least half way around the pipe, and not obstruct the text. If labeling inside equipment, the size of the labels may be adjusted, provided they remain easily seen and readable. Consideration should be given to using tags instead of labels.

NOTE: Labeling sizes for piping 19 mm (0.75 inch) or larger in outside diameter were taken directly from ANSI A13.1. Smaller sizes were developed for this guide.

6.7 The height of the label should be at least two times (2×) the height of the text, with the text centered in the label. The label should allow a length of background color on each end of the text at least one half pipe diameter in length but not less than 12.7 mm (0.5 inch).

6.8 The orientation of text on the label should conform to local customs, conventions, or practices.

6.9 Directional flow arrows should be applied to all piping systems indicating the direction of material flow within the system. The arrow(s) should be included as part of the primary label but may be applied separately within close proximity to the primary label (e.g., less than 5 cm (2 inches) for pipe diameters up to 5 cm (2 inches); less than 15 cm for larger pipe diameters).

6.10 Labels should be applied to the piping system at the following minimum locations:

- On both sides of a barrier penetration (i.e., walls, floors, ceilings, equipment enclosures, etc.). The labels should be applied within 1 meter (3.3 feet) or as close as practical to the barrier.
- As close as practical to all changes in direction where the length of run in the new direction is 1 meter (3.3 feet) or greater. Where several directional changes are made within a short length of run, labels need not be applied at every change in direction, but should be applied at sufficient intervals to readily identify the piping.
- On long runs, the labels should be applied at intervals not exceeding 6 meters (20 feet). Shorter intervals should be used for small diameter pipe and where several pipes are run in close proximity.
- Where several pipes are run parallel to each other, the labeling for all pipes should be in line with each other (i.e., should not be staggered).
- A sufficient number of labels should be applied at each location to ensure that they can be readily seen and read from all viewing or approach direction(s).
- Each side of a “T” connection should be labeled within 1 meter (3.3 feet) of the “T”.

Table 2 Recommended Label and Text Sizes for Various Piping Diameters

<i>Outside Diameter of Pipe or Covering</i>		<i>Minimum Length of Label</i>		<i>Minimum Height of Letters</i>	
<i>Millimeters</i>	<i>Inches</i>	<i>Millimeters</i>	<i>Inches</i>	<i>Millimeters</i>	<i>Inches</i>
Less than 13	Less than 0.5	51	2	3	0.125
13–19	0.5–0.75	102	4	6	0.25
19–38	0.75–1.5	203	8	13	0.5
38–64	1.5–2.5	203	8	19	0.75
64–203	2.5–8	305	12	32	1.25
203–254	8–10	610	24	64	2.5
Over 254	Over 10	813	32	89	3.5

6.11 Where it is impractical to attach markings or labels directly to the pipe, a tag may be used. The tag should be durable for the environment in which it is used and should be securely attached to the piping system such that it cannot be easily removed or relocated from its installed position. The tag or tags should contain the same information as defined for labels in Sections 6.4 and 6.5. Standard color-coding for tags should be the same as defined for labels in Table 1. Tags should be installed at the same locations as required for other identification methods defined in Section 6.10.

7 Related Documents

7.1 ANSI Document

ANSI Z535.1 — Safety Color Code

7.2 BSI Document²

BS 1710 — Identification of Pipelines and Services

7.3 DIN Document³

DIN 2403 — Kennzeichnung von Rohrleitungen nach dem Durchflußstoff (Identification Code for Pipe Lines According to Media)

7.4 ISO Documents⁴

ISO 3864 — Safety Colours and Safety Signs

ISO/DIS 14726-1 — Ships and Marine Technology — Identification Colours for the Contents of Piping Systems — Part 1: Main Colours and Media (Revision of ISO/R 508:1966) { ISO/R 508:1966 was withdrawn by TC 8 in 1986 }

² British Standards Institution, 389 Chiswick High Road, GB-London W4 4AL, Telephone:+ 44.181.996.90.00, Telefax:+ 44.181.996.74.00, E-mail: info@bsi.org.uk, WWW: <http://www.bsi.org.uk/>

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⁴ International Organization for Standardization, Central Secretariat, 1 rue de Varembe, Case postale 56, CH-1211, Geneve 20, Switzerland, Telephone:+ 41.22.749.01.11, Fax:+ 41.22.733.34.30, E-mail: central@iso.ch, WWW: <http://www.iso.ch/>

7.5 United States Department of Transportation
Document⁵

Title 49, Code of Federal Regulations

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⁵ US Government Printing Office, Washington, D.C., USA

SEMI F35-0304

TEST METHOD FOR ULTRA-HIGH PURITY GAS DISTRIBUTION SYSTEM INTEGRATION VERIFICATION USING NON-INVASIVE OXYGEN MEASUREMENT

This test method was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on December 4, 2003. Initially available at www.semi.org February 2004; to be published March 2004. Originally published September 1998.

1 Purpose

1.1 This test method defines a procedure to monitor the integrity of ultra-high purity (UHP) gas distribution systems by detecting the ingress of atmospheric oxygen. This test method would be used to evaluate an “active” UHP gas distribution system on a continuous basis by using *non-invasive* O₂ measurement, without requiring an interruption of the process tools using the UHP gases of interest.

1.2 This test method should be used to protect semiconductor fabrication processes using the UHP gases, which may be sensitive to contamination by any of the common atmospheric impurities, such as N₂, O₂, H₂O, CO₂, before product yield problems develop.

1.3 This is the first such test method that describes a noninvasive leak detection and locating procedure. It differs from SEMI F1, which is an invasive technique for identifying leak sources using a mass spectrometer and a helium tracer gas.

2 Scope

2.1 This test method applies to UHP gas distribution systems used in semiconductor manufacturing facilities and comparable research and development areas.

2.2 This test method applies to bulk gas distribution systems carrying UHP gases such as N₂, Ar, He, H₂, N₂O, SF₆, and many halocarbons. In most cases, O₂ is present only in ultra-low trace levels (typically less than 1.0 ppb).

2.3 This test method will provide real-time monitoring of UHP gas distribution systems, resulting in meaningful system integrity verification, atmospheric contaminant trending analysis, and leak locating.

2.4 This test method will provide the user with sufficient information to identify and troubleshoot sources of atmospheric leakage into the UHP gas distribution system.

2.5 This test method includes the specification of the required O₂ analytical equipment, standard methods for proper use of the O₂ analytical equipment, and

manipulation of the O₂ data in identifying atmospheric leak sources.

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

3 Limitations

3.1 This test method will only be successful in identifying the presence and location of leaks from atmospheric air using O₂ as the tracer gas representative of all atmospheric air contaminants (i.e., N₂, O₂, H₂O, CO₂). There is no capability to identify cross-contaminant species between different UHP gases which only contain ultra-low trace levels of O₂ or outward leaks to atmosphere.

3.2 This test method will not focus specifically on the process of detecting the exact location of every possible atmospheric air leakage source. Rather, it will describe techniques and examples demonstrating how atmospheric air leaks can be reliably identified and located, in general.

3.3 Results from this test method may not ensure that other common atmospheric impurities (e.g., H₂O) are below their required specification limits after a leak has been identified, corrected, and O₂ levels have returned to normal. For example, since O₂ diffuses more quickly and has a weaker surface adsorption than H₂O, O₂ will be the first atmospheric impurity detected. However, H₂O will likely be the last atmospheric impurity to be purged out once a leak has been identified and repaired.

3.4 This test method is not a direct substitution for SEMI F1.

4 Referenced Standard

4.1 SEMI Standard

SEMI F1 — Specification for Leak Integrity of High Purity Gas Piping Systems and Components

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *ppbv* — parts per billion by volume.

5.2 Definitions

5.2.1 *atmospheric impurities* — the common impurities to UHP gases that are found in atmospheric air.

NOTE 1: Impurities such as N₂, O₂, H₂O, CO₂.

5.2.2 *contaminant signature* — typical baseline performance of a specific sample point within a large distribution system with respect to its O₂ impurity concentration. This is considered to be a normal and acceptable impurity level.

5.2.3 *critical sample point* — gas sample point which is monitored continuously because it is deemed in a critical location in the distribution system, or that which is most sensitive to causing product/process quality problems from the ingress of atmospheric air impurities.

5.2.4 *investigative sample point* — gas sample point which is selected for spot checking analysis as part of an investigation to locate an atmospheric air leak source.

5.2.5 *low detectable limit* — defined as the smallest level of O₂ measurement which can be quantified after performing a blank test on zero gas. For our purposes, this is determined to be 2 times the total peak-to-peak noise over 8 hours while the O₂ analyzer is sampling on zero gas.

5.2.6 *point of use* — the connection point where the gas distribution system connects to the process tool which uses the UHP gas.

5.2.7 *sensitivity* — Defined as the smallest detectable step change in O₂ that the analyzer is capable of sensing.

5.2.8 *ultra-high purity (UHP) gas distribution system* — semiconductor fab gas delivery system which typically contains impurities from atmospheric air of < 0.2 ppb at the input source, and atmospheric impurities at the point-of-use of typically < 1 ppb.

5.2.9 *weekly zero drift* — total analyzer drift while measuring zero gas over a 1-week time period. Includes all components of analyzer drift.

5.2.10 *zero gas* — test gas which is known to contain less than 0.1 ppb of O₂ as an impurity.

6 Summary of Method

6.1 The user must identify critical sampling locations in the UHP gas distribution system. These points are either specific process tool points-of-use or key lateral branch lines, which must avoid atmospheric contamination, or representative of a critical purity zone containing process tools that are particularly sensitive to atmospheric contaminants.

6.2 Connect O₂ analysis equipment to these critical sample points for continuous monitoring.

6.3 Qualify the performance of the O₂ analysis equipment before collecting data.

6.4 Begin continuous monitoring of critical sample points. Identify typical or baseline “contaminant signature” performance of the UHP gas distribution system.

6.5 Use continuous trend data to identify problematic events (contaminant spikes) and/or significant changes from the “contaminant signature” performance level.

6.6 Use additional investigative sample point measurements (spot checks) along with observable correlations to gas distribution system usage to locate and correct atmospheric leak sources.

7 Interferences

7.1 While sampling from any point in the gas distribution system, it is critical that the sample be delivered to the O₂ analytical equipment without introducing additional atmospheric O₂. This could create significant errors in the analysis. Observing a higher O₂ reading after reducing the sample flow to the analyzer may be an indication that there is a leak between the sample point and the O₂ analysis equipment.

7.2 An O₂ analyzer may become inaccurate due to improper calibration or simply due to long-term analyzer drift. This will cause inaccurate O₂ readings which do not represent the true performance of the UHP gas distribution system. The O₂ analyzer must be operated per the manufacturer’s recommendation for calibration technique, calibration frequency, and other routine maintenance.

7.3 Some O₂ analytical methods have cross-sensitivity to other gaseous components which may be found in UHP gases (i.e., H₂, CO, CH₄, and other hydrocarbons).

8 Requirements

8.1 *Personnel Qualification* — Personnel performing these tests in accordance with this test method shall have suitable training and experience. Such personnel shall, as a minimum, be knowledgeable of:

8.1.1 The operation and calibration of the specific equipment used in performing this test.

8.1.2 The proper procedures in handling UHP gases as agreed upon by supplier and user.

8.1.3 The proper safety procedure in handling combustible or toxic gases as agreed upon by supplier and user.

8.1.4 The behavior of atmospheric leak sources relative to position, pressure/flow, and gas usage.

9 Apparatus

9.1 An analyzer must be selected which is capable of continuous ultra-low trace O₂ measurements, with the ability to report measurements with at least once per minute frequency.

9.2 The ultra-low trace O₂ analyzer shall meet or exceed the following specifications. These specifications are appropriately defined in Section 5:

Sensitivity	< 0.1 ppb
Low Detectable Limit	< 0.3 ppb
Weekly Zero Drift	< 0.5 ppb
Response Time	< 10 minutes for 90% of a 0–1 ppb O ₂ step change

NOTE 2: The O₂ analysis must be conducted using an analytical method which meets or exceeds the specifications required by this test method. If not, the user will not be able to confidently distinguish true gas distribution system impurity trends from false analyzer trends.

10 Safety Precautions

10.1 This test method is not a replacement for safety regulations. It is the responsibility of the user to ensure that the UHP gas distribution systems under analysis comply with applicable safety regulations, as agreed to between gas supplier and user.

10.2 It is also the responsibility of the user to comply with applicable safety regulations governing the operation of the required O₂ analytical equipment, as specified by the analyzer manufacturer.

11 Identify Sample Point Locations

11.1 Identify the critical sample point locations in the UHP gas distribution system which allow for a continuous gauging of overall system integrity. These shall include, at a minimum:

11.1.1 An exit purity O₂ measurement of the UHP gas at the furthest point of the gas distribution system within the fab.

11.1.2 Sample point locations representing subsections of the UHP gas distribution system, which are deemed critical for the avoidance of atmospheric impurities.

11.1.3 Point-of-use process tool locations which are deemed critical for the avoidance of atmospheric impurities.

11.2 A source purity O₂ measurement of the UHP gas entering the gas distribution system is suggested to be used as a reference comparison against other points within the fab.

11.3 Additional investigative (spot check) O₂ measurements may be required at sample point locations which aid in deducing the location of suspected atmospheric leak sources. This will vary from situation to situation. (See Section 16.)

11.4 The exact sample tap location must be representative of the measurement point of interest. Avoid measurement of dead-leg locations.

12 Preparation of Sample Point Locations

12.1 Thoroughly pre-purge the sample point prior to connecting the O₂ analyzer.

12.2 Thoroughly cycle-purge any regulators or other components in the sample system. Cycle the pressure at least 20 times with a high/low pressure ratio of at least 5 times.

12.3 Connect the O₂ analyzer to the sample point using appropriate UHP gas lines and fittings. Avoid using long lengths of bellows-type tubing because of long purge-down requirements. Avoid using Teflon or plastic tubing of any kind. Teflon and most other plastics are extremely permeable to O₂ and will cause atmospheric O₂ leakage. Only a few feet of plastic tubing can introduce ppm levels of O₂.

12.4 Establish flow from the sample point to the O₂ analyzer per the manufacturer's recommendations.

13 Calibration and Qualification

13.1 Follow the manufacturer's recommendations for initial start-up and calibration of the O₂ analyzer.

13.2 After initial start-up and calibration, adhere to the manufacturer's recommendations for routine calibration and maintenance needed to achieve the required performance specification as described in Section 9.2.

13.3 There will be two modes of O₂ measurement:

13.3.1 *Critical Sample Point O₂ Measurement*

13.3.1.1 Applicable to O₂ measurements performed continuously on the critical sample point locations identified earlier.

13.3.1.2 Prior to continuous data collection use of the analyzer, validate it by demonstrating the capability required in the specification for Weekly Zero Drift. Chart the zero baseline drift and peak-to-peak noise while on zero gas for 1 week.

13.3.1.3 Re-validate the O₂ analyzer performance by running a 24-hour blank test on zero gas monthly.

13.3.2 *Investigative Sample Point O₂ Measurement*

13.3.2.1 Applicable to spot check O₂ measurements performed in the investigation of locating atmospheric leak sources.

13.3.2.2 The O₂ analyzer must be operated for a time period sufficient to establish the “contaminant signature” at the point of interest.

13.3.2.3 Validate the O₂ analyzer with a blank test prior to each use. Chart zero baseline drift over a period of time which is twice as long as the expected time required to make the O₂ measurement. If a measurement can typically be made successfully in less than one hour, then verify the zero drift and peak-to-peak noise with a two-hour blank test.

14 Data Collection Procedure

14.1 Collect all O₂ measurement data at a minimum of 1 point/minute using a chart recorder or data acquisition device.

14.2 Graph data points with sufficient resolution to observe trends over a one-week period. The graph must also be able to resolve O₂ spikes which may last only 10–15 minutes.

15 Interpretation of Results

15.1 Generate graphs of the baseline “contaminant signature” for each critical sample point location. Compare the baseline “contaminant signature” with the O₂ analyzer’s blank run on zero gas to validate that the analyzer is producing useful data. The base-line “contaminant signature” should exhibit equal or greater O₂ variation than the blank run on zero gas.

15.2 Problem Identification

15.2.1 Observe data from critical sample point locations and look for deviations (abnormal trends or events) from the baseline “contaminant signature”.

15.2.2 Determine if deviations (observed trends or events) are sufficient to impede mandatory atmospheric impurity specifications or influence critical product/process quality.

15.2.2.1 If yes, proceed into Section 16.

15.2.2.2 If no, continue observing O₂ data from critical sample point locations.

16 Atmospheric Leak Locating Procedure

16.1 Familiarize yourself with Figure 1 before proceeding with the remainder of Section 16.

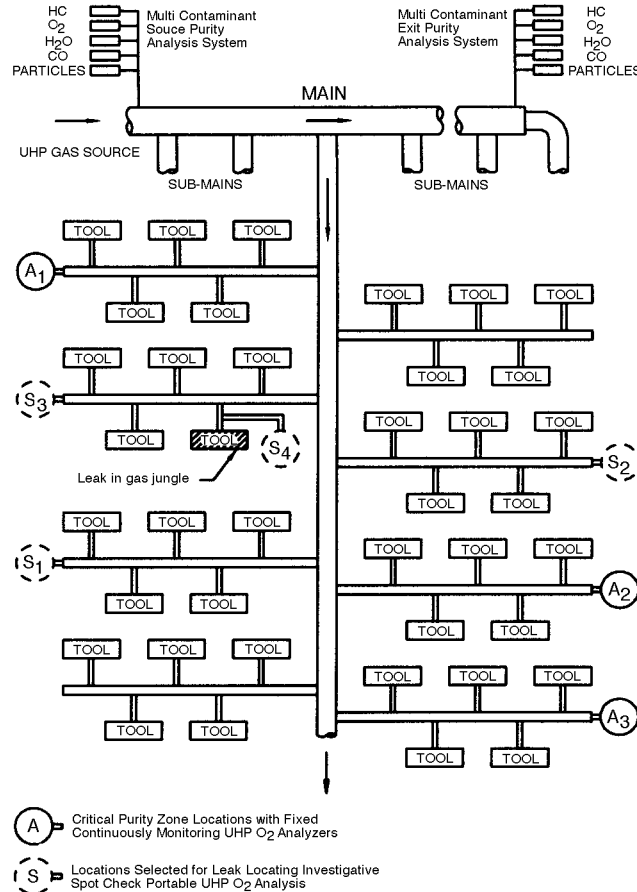


Figure 1
Typical Fab UHP Gas Distribution System

16.2 Isolate a zone where the contamination is likely to originate from by comparing the O₂ levels at the critical continuous monitoring sample point locations. Assume the leak source is somewhere between the last uncontaminated sample point and the first contaminated sample point going downstream. Some knowledge of the gas distribution flow path will be required here.

16.3 Compare the data from the O₂ analyzer detecting the contamination with pressure and flow data, or gas usage demand over time in the specific lateral of the gas distribution system where the O₂ analysis is being made.

16.4 Correlate the changes in O₂ readings which show abnormally high O₂ levels with specific tool cycles that cause the UHP gas pressure and flow in the lateral line to change. The timing of the analyzer's response to these usage demands will give some clues as to the approximate position of the atmospheric leak source.

NOTE 3: Back-contamination is likely originating in a leg which is dead at the time the O₂ analyzer responds. Actual response time will depend on the time needed for the contaminant to travel to the O₂ analyzer plus the response time of the O₂ analyzer.

16.5 If no demand cycles in the lateral cause O₂ readings to change, then the source of contamination is likely to occur further upstream in the distribution system, prior to the intersection of the lateral in question. This assumes that there is always a net flow of gas in the line leading to the intersection of the lateral being checked.

16.6 Perform an investigative O₂ measurement (spot check) with a portable O₂ analyzer at each lateral location that is suspected in causing contamination in the main or sub-main line which supplies the lateral where the contamination was originally detected. Work upstream away from the lateral which showed a sign of contamination.

16.7 Once the lateral with the contamination source has been identified, use the steps outlined in Sections 16.3 and 16.4 to locate the exact point of the atmospheric leak source.

16.8 Refine the selection of investigative sample point locations along with a careful study of system flow dynamics until the leak is pinpointed and repaired.

16.9 Confirm that the leak source has been corrected by providing an investigative O₂ measurement downstream of the leak source.

17 Example in Locating Leak Source

17.1 See Figure 1 which is a schematic representation of a typical fab branching type gas distribution system. Since a loop type gas distribution system has one less piping level between the main line and the tools, it is more straightforward to monitor and diagnose. This example, although more complex, has been demonstrated in practice. It shows how an atmospheric leak originating at a single tool location can back-contaminate a sub-main distribution line, yet not contaminate all the way back to the main line. In this example, the contamination would not be detected by the UHP analysis instrumentation typically installed on the main line.

17.2 The “contaminant signature” oxygen analyzers are installed at critical purity locations, shown in Figure 1 as A₁, A₂, and A₃. These analyzers show baseline “contaminant signature” O₂ levels of 0.2–0.5 ppb. By definition, as established by the user for each critical purity location, these are normal contaminant levels at these locations.

17.2.1 A₂ and A₃ begin showing excursions up to 1–2 ppb that last for several minutes, then settle back into the normal 0.2–0.5 ppb range. The excursions take place infrequently at first, but then develop some regularity. Although the absolute O₂ level detected does not yet indicate that the potential atmospheric leak (H₂O and N₂ included) would pose a direct risk to the critical purity of process tools located nearby, it does differ significantly from the baseline “contaminant signature”. This prompts an investigation before a full-scale process production problem results.

17.2.2 Each of the lateral lines being monitored by A₂ and A₃ shows the same level of O₂ contamination, so it is deduced that the contamination is coming from the sub-main line which feeds both laterals. Since analyzer A₁ does not show the contamination, it is further deduced that the leak source is located between A₁ and A₂.

17.3 Working back upstream along the sub-main, investigative O₂ measurements are made at each lateral location ahead of the A₂ lateral. These investigative O₂ measurements are made using a portable O₂ analyzer per Sections 12 and 13.

17.4 At S₁ and S₂, similar O₂ contamination events are observed as compared with points A₂ and A₃. Further, no lateral line flow changes (due to tool gas demand cycles in those laterals) cause any change in the O₂ level.

17.5 At the third lateral, sampled as S₃ in Figure 1, the O₂ level is significantly higher (ranging between 5–10 ppb), with periodic spikes of 50–100 ppb. At this point, it is observed that when the usage in lateral S₃ drops to zero (a dead leg condition), analyzers A₂ and A₃ show O₂ readings above the “contaminant signature”. It is therefore deduced that the S₃ lateral is the source of the atmospheric leak.

17.6 By correlating the O₂ data at S₃ with the gas usage from the tools on this lateral, the leak source can be further traced within this lateral. (Refer to Figure 2.)

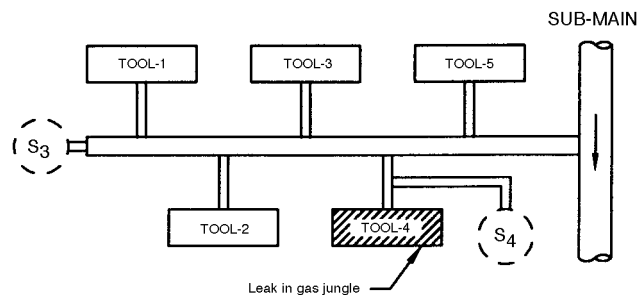


Figure 2
Branch UHP Gas Line with Ambient Air Leak Contaminating Sub-Main Line

17.6.1 When Tool-1, Tool-2, or Tool-3 demands gas, the O₂ reading drops in proportion to the total flow of gas in the lateral line. The higher flow rates act to dilute the effects of the leak source and thus lower the O₂ reading at S₃.

17.6.2 It is observed that when Tool-5 demands gas, there is no change in the O₂ reading at S₃. Therefore, it is deduced that the leak source is located further out along the lateral line.

17.6.3 When Tool-4 demands gas, it causes a more significant drop in the O₂ reading at S₃. Further, it is observed that the time required for the analyzer to respond after Tool-4 begins demanding gas correlates to the time needed for the analyzer's sample flow alone to purge down the lateral line volume up to the analyzer, when Tool-1, -2, and -3 are not demanding gas.

17.6.4 This deductive inference technique pinpoints the leak source to the Tool-4 lateral line.

17.6.5 After Tool-4 has stopped demanding gas (yet before any other Tools on this lateral demand gas), the O₂ reading at S₃ continues to drop gradually over several minutes. It then gradually begins to elevate once again. This indicates that the actual leak is not very close to the connection of Tool-4 at the lateral line. It suggests more that the leak source is closer to the tool itself.

17.7 A final investigative sample point is selected along the line leading to Tool-4. This is shown as S₄ in Figure 2. It is observed that the O₂ readings drop to 0.2–0.5 ppb when Tool-4 demands gas. S₄ begins to show an O₂ elevation after the Tool-4 stops demanding gas. This concludes that the leak source is originating from Tool-4 itself.

18 Related Documents

18.1 SEMI Standard

SEMI E49.8 — Guide For High Purity And Ultrahigh Purity Gas Distribution Systems In Semiconductor Manufacturing Equipment

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

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SEMI F36-0299 (Reapproved 1104) GUIDE FOR DIMENSIONS AND CONNECTIONS OF GAS DISTRIBUTION COMPONENTS

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 1993; last published February 1999.

1 Purpose

1.1 The purpose of this guide is standardization of dimensional and mechanical/electrical connection attributes for components used within gas distribution systems. Standardization of these component interfaces will allow for interchangeability of gas distribution system components. Filters/purifiers, shut-off valves, pressure regulators, MFC/MFMs, and pressure transducers are the types of gas distribution components that would benefit from being interchangeable.

2 Scope

2.1 This guide is for use with 1/4-inch distribution systems at operating pressures no greater than 345 kPa (50 psi).

2.2 The guide includes the following items:

2.2.1 Physical characteristics specific to each component. These include a generalized drawing of the component, specific end-to-end lengths for each type of end connection, maximum envelope dimensions, base-to-centerline dimensions, tolerances, and bottom mounting information.

2.2.2 Electrical connections specific to each component, where applicable. These include pin-outs and types of connectors.

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

3 Limitations

3.1 This guide is not intended to dictate how to build a gas distribution system, but to allow for interchangeability of components.

3.2 This standard does not include recommendations for sensor-bus compliant interfaces.

3.3 This standard does not apply to surface mount components used in modular type gas systems.

4 Referenced Standards

4.1 None.

5 Terminology

5.1 Definitions

5.1.1 *component* — an individual piece or a complete assembly of individual pieces capable of being joined with other pieces or components.

5.1.2 *filter* — a porous device, generally constructed of polymer, metal, or ceramics and housed in a metal chamber, which traps particles, preventing them from being transported downstream.

5.1.3 *mass flow controller* (MFC) — a self-contained device (consisting of a transducer, control valve, and control and signal-processing electronics) commonly used in the semiconductor industry to measure and regulate the mass flow of gas.

5.1.4 *mass flow meter* (MFM) — a self-contained device, consisting of a mass flow transducer and signal-processing electronics, commonly used in the semiconductor industry to measure the mass flow of gas.

5.1.5 *pressure regulator* — a valve designed to reduce a high incoming pressure (for example, from a cylinder) to a lower outlet pressure by automatically opening to allow flow until a desired, preset pressure on the outlet side is reached, then automatically throttling closed to stop further pressure increase.

5.1.6 *pressure transducer* — a component which mechanically or electrically senses gas pressure. It typically consists of a sensor and signal-processing electronics which enables remote indication of gas pressure.

5.1.7 *purifier* — an in-line device used for the removal of homogeneous impurities from gases, typically consisting of a packed-bed of active solids contained in a stainless steel housing. The active purification media may remove impurities such as moisture, oxygen, CO, CO₂, hydrocarbons, hydrogen, or nitrogen from specific gases using a variety of chemical reaction, physisorption, or chemisorption mechanisms. Point-of-use purifiers often contain a particle filter within the same housing.

5.1.8 *valve* — a device that controls the flow or pressure of a gas. Valve functions can include shutoff, metering, backflow prevention, and pressure relief.

6 Ordering Information

6.1 Device manufacturers may use this guide when procuring processing equipment to communicate to the equipment supplier the interface specifications required for interchangeability of components. This document may also be used by semiconductor processing equipment suppliers to specify standardized interfaces to component and module suppliers.

6.2 Orders for components in accordance with this standard shall include:

6.2.1 This standard number and date of issue.

6.2.2 Reference to the table number.

6.2.3 Reference to the option number, if applicable.

7 Filters and Purifiers

7.1 Filters less than 30 SLM and purifiers less than 1 SLM should conform to the interface design recommendations appearing in Figure 1 and Table 1.

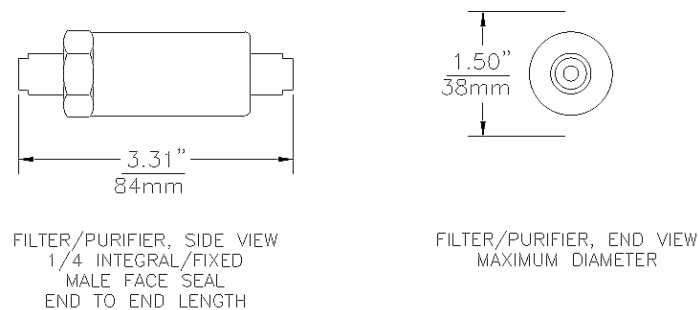


Figure 1
Filter and Purifier Interface Design



Table 1 Filter and Purifier Interface Recommendations

	<i>Filter and Purifier</i>
End-to-End Lengths: 1/4-inch Integral/fixed male face seal 1/4-inch Swivel face seal (male or female) 1/4-inch Tube weld NOTE 1: The tolerance on length for machined components is ± 0.15 mm (0.006 in.) from the component centerline to each end. NOTE 2: The tolerance on length for welded components is ± 0.5 mm (0.020 in.) from the component centerline to each end.	84 mm (3.31 in.) - -
Overall Component Envelope: Maximum Diameter	38 mm (1.5 in.)
Base to Centerline of Flow Path:	-
Mounting provisions: Threaded holes Pattern	None required -
Fittings: Process	1/4-inch
Control:	None required

8 Shut-off Valves

8.1 Shut-off valves should conform to the interface design recommendations appearing in Figure 2 and Table 2.

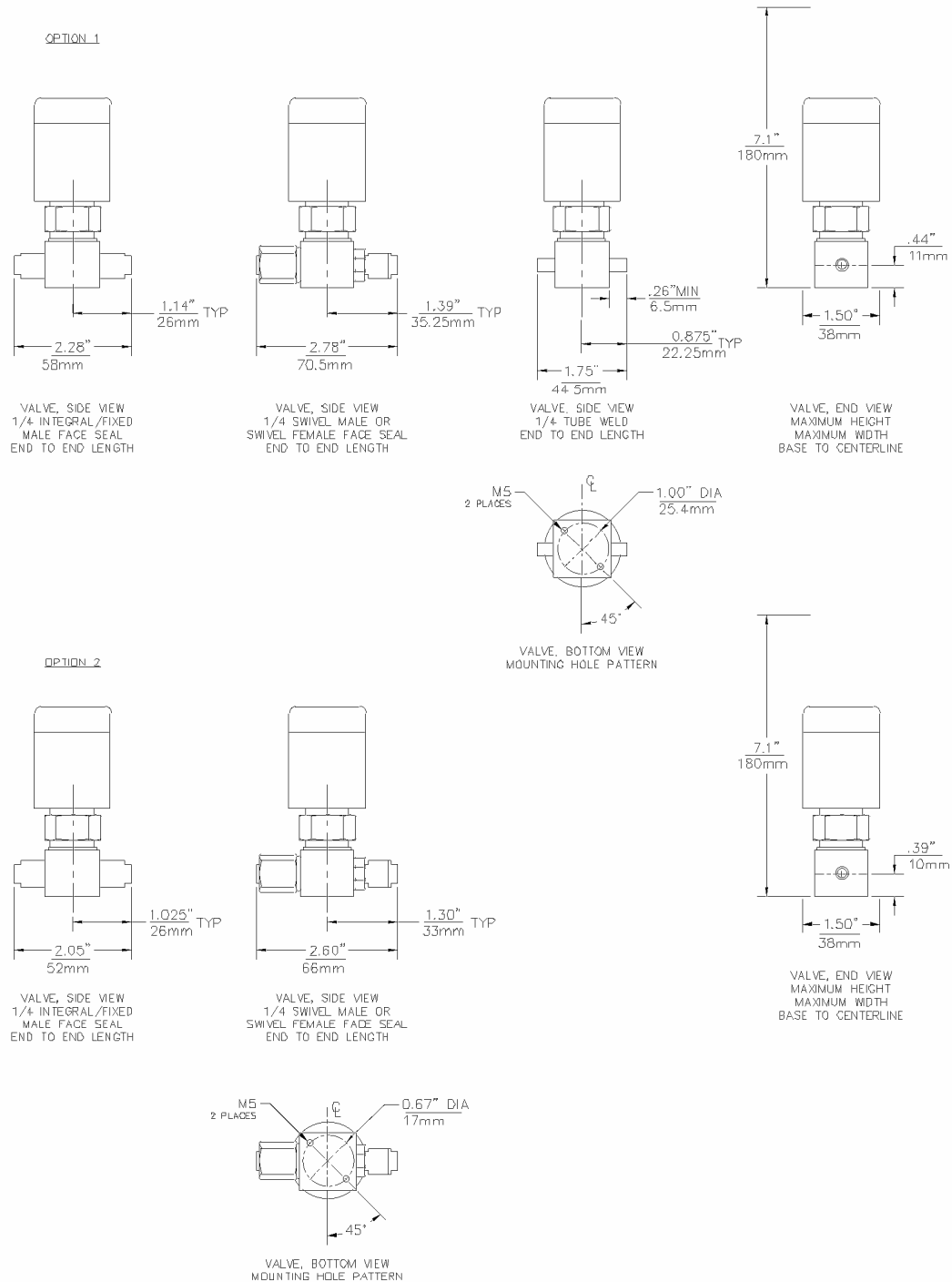


Figure 2
Shut-off Valve Interface Design

Table 2 Shut-off Valve Interface Recommendations

	<i>Option 1 Shut-off Valves</i>	<i>Option 2 Shut-off Valves</i>
End-to-End Lengths: 1/4 inch Integral/fixed male face seal 1/4 inch Swivel face seal (male or female) 1/4 inch Tube weld	58 mm (2.28 in.) 70.5 mm (2.78 in.) 44.5 mm (1.75 in.)	52 mm (2.05 in.) 66 mm (2.60 in.) -
NOTE 1: The minimum length for each tube weld stub should be 6.5 mm (0.26 in.). The tolerance on length for machined components is ± 0.15 mm (0.006 in.) from the component centerline to each end. The tolerance on length for welded components is ± 0.50 mm (0.020 in.) from the component centerline to each end.		
Overall Component Envelope: Maximum height Maximum width (Excluding lever and toggle handles)	180 mm (7.1 in.) 38 mm (1.5 in.)	
Base to Centerline of Flow Path:	11 mm (0.44 in.) ± 0.3 mm (0.012 in.)	10 mm (0.39 in.) ± 0.3 mm (0.012 in.)
Mounting Provisions: Threaded holes Pattern	M5 \times 0.8 – 6H; 6 mm (0.24 in.) perfect thread depth 25.4 mm (1.00 in.) bolt circle centered on bottom of base with two threaded holes 180° apart and 45° off of the vertical or horizontal axis. One hole in each of the upper left and lower right quadrants when looking at the bottom of the component.	M5 \times 0.8 – 6H; 5mm (0.20 in.) perfect thread depth 17 mm (0.67 in.) bolt circle centered on bottom of base with two threaded holes 180° apart and 45° off of the vertical or horizontal axis. One hole in each of the upper left and lower right quadrants when looking at the bottom of the component.
NOTE 2: The M5 \times 0.8 threaded mounting holes will accept 10–32 screws, but 10–32 threaded holes will not accept M5 \times 0.8 screws.		
Fittings: Process	1/4-inch	
Control:	Manual and Pneumatic	

9 Pressure Regulators

9.1 Pressure regulators should conform to the interface design recommendations appearing in Figure 3 and Table 3.

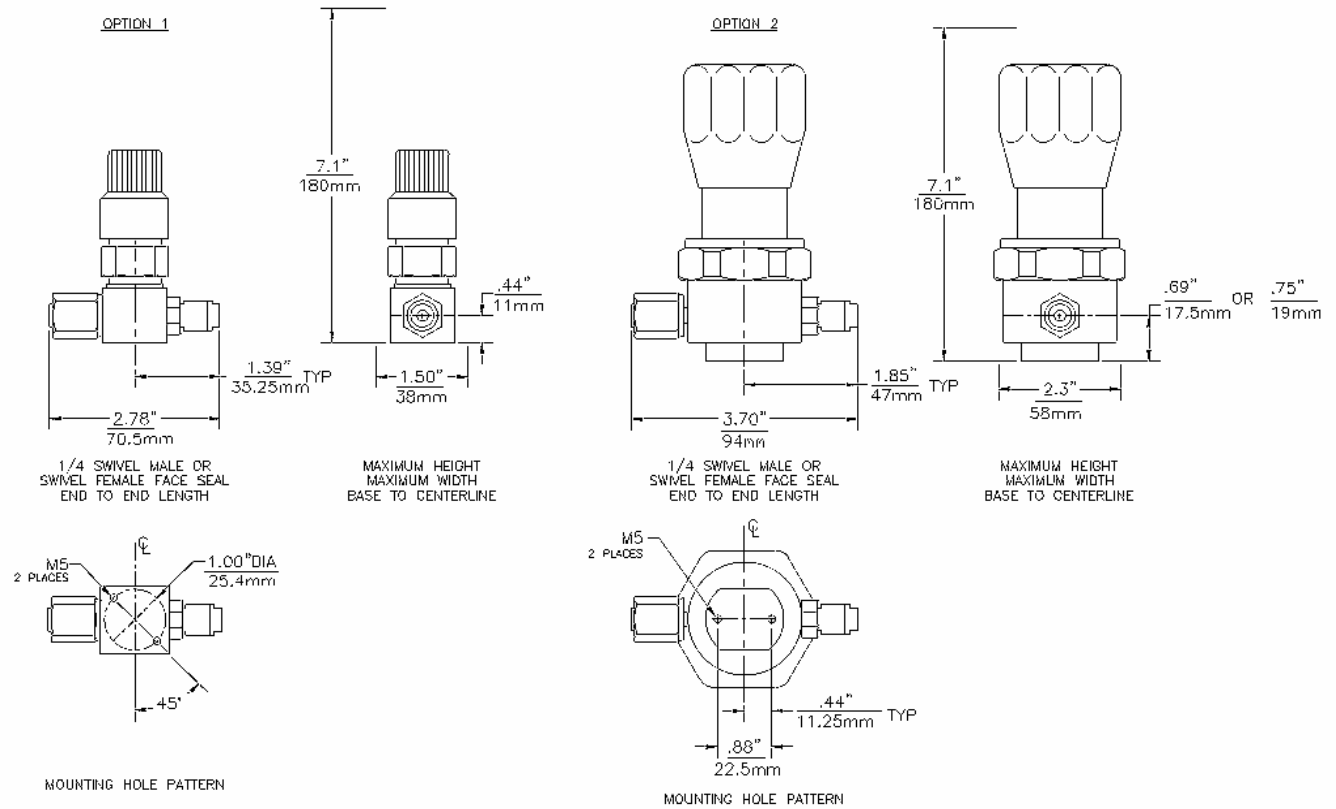


Figure 3
Pressure Regulator Interface Design

Table 3 Pressure Regulator Interface Recommendations

	<i>Option 1 Pressure Regulators</i>	<i>Option 2 Pressure Regulators</i>
End-to-End Lengths: 1/4-inch Integral/fixed male face seal 1/4-inch Swivel face seal (male or female) 1/4-inch Tube weld	- 70.5 mm (2.78 in.) -	- 94 mm (3.70 in.) -
NOTE 1: The tolerance on length for machined components is ± 0.15 mm (0.006 in.) from the component centerline to each end. The tolerance on length for welded components is ± 0.50 mm (0.020 in.) from the component centerline to each end.		
Overall Component Envelope: Maximum height Maximum width	180 mm (7.1 in.) 38 mm (1.5 in.)	180 mm (7.1 in.) 58 mm (2.3 in.)
Base to Centerline of Flow Path:	11 mm (0.44 in.) ± 0.3 mm (0.012 in.)	17.5 mm (0.69 in.) ± 0.3 mm (0.012 in.) or 19 mm (0.75 in.) ± 0.3 mm (0.012 in.)
Mounting Provisions: Threaded holes Pattern	M5 \times 0.8 – 6H; 6 mm (0.24 in.) perfect thread depth 25.4 mm (1.00 in.) bolt circle centered on bottom of base with two threaded holes 180° apart and 45° off of the vertical or horizontal axis. One hole in each of the upper left and lower right quadrants when looking at the bottom of the component.	M5 \times 0.8 – 6H; 6 mm (0.24 in.) perfect thread depth Two threaded holes, centered on bottom of base, in line with flow path, spaced 22.5 mm (0.88 in.) between centers
NOTE 2: The M5 \times 0.8 threaded mounting holes will accept 10–32 screws, but 10–32 threaded holes will not accept M5 \times 0.8 screws.		
Fittings: Process	1/4-inch	
Control:	Manual	

10 MFCs and MFMs

10.1 MFCs and MFMs should conform to the interface design recommendations appearing in Figure 4 and Table 4.

10.2 A nine pin connector was specified along with a signal for each pin to allow for MFC/MFM interchangeability. If additional signals are required, an additional connector should be specified. If signals are different, an alternate connector should be specified.

10.3 Current technology limits the MFC and MFM base-to-centerline dimension to 12.7 mm (0.50 in.). If future technologies permit, 11 mm (0.44 in.) should be considered as an option for the base-to-centerline dimension. This recommendation would improve modularity by permitting selection of each component type with a common base-to-centerline dimension.

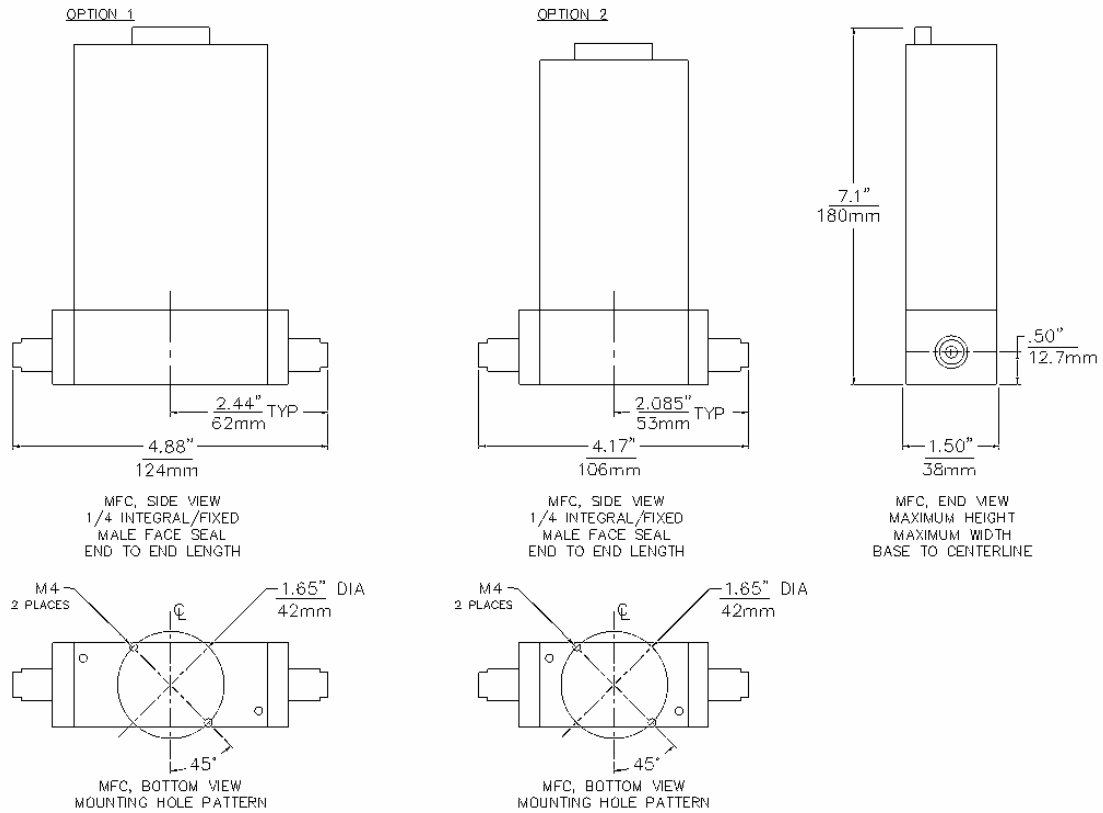


Figure 4
MFC and MFM Interface Design

Table 4 MFC and MFM Interface Recommendations

	<i>Option 1 MFC and MFM</i>	<i>Option 2 MFC and MFM</i>
End-to-End Lengths: 1/4-inch Integral/fixed male face seal 1/4-inch Swivel face seal (male or female) 1/4-inch Tube weld	124 mm (4.88 in.) - -	106 mm (4.17 in.) - -
NOTE 1: The tolerance on length is ± 0.50 mm (0.020 in.) from the component centerline to each end.		
Overall Component Envelope: Maximum height Maximum width	180 mm (7.1 in.) 38 mm (1.5 in.)	
Base to Centerline of Flow Path:	12.7 mm (0.50 in.) ± 0.3 mm (0.012 in.)	
Mounting provisions: Threaded holes Pattern	M4 \times 0.7 – 6H; 3 mm (0.12 in.) minimum perfect thread depth 42 mm (1.65 in.) bolt circle centered on bottom of base with two threaded holes 180° apart and 45° off of the vertical or horizontal axis. One hole in each of the upper left and lower right quadrants when looking at the bottom of the component.	
Fittings: Process	1/4-inch	
Control:	Electrical 9-pin sub-D connector Analog signal pin out as follows:	
	Pin 1.	Valve Closed (Ground Pin to Close Valve)
	Pin 2.	0–5 V _{DC} Flow Signal Output
	Pin 3.	+15 V _{DC} Power Supply
	Pin 4.	Power Common
	Pin 5.	-15 V _{DC} Power Supply
	Pin 6.	0–5 V _{DC} Setpoint Input
	Pin 7.	Output Common
	Pin 8.	Setpoint Common
	Pin 9.	Valve Test Point

11 Pressure Transducers

11.1 Pressure transducers should conform to the interface design recommendations appearing in Figure 5 and Table 5.

11.2 To improve modularity of gas distribution systems the following recommendation should be considered for future pressure transducer design. This recommendation would match the pressure transducer interface design to the dimensions of both the shut-off valve Option 1 found in Table 2 and pressure regulator Option 1 found in Table 3.

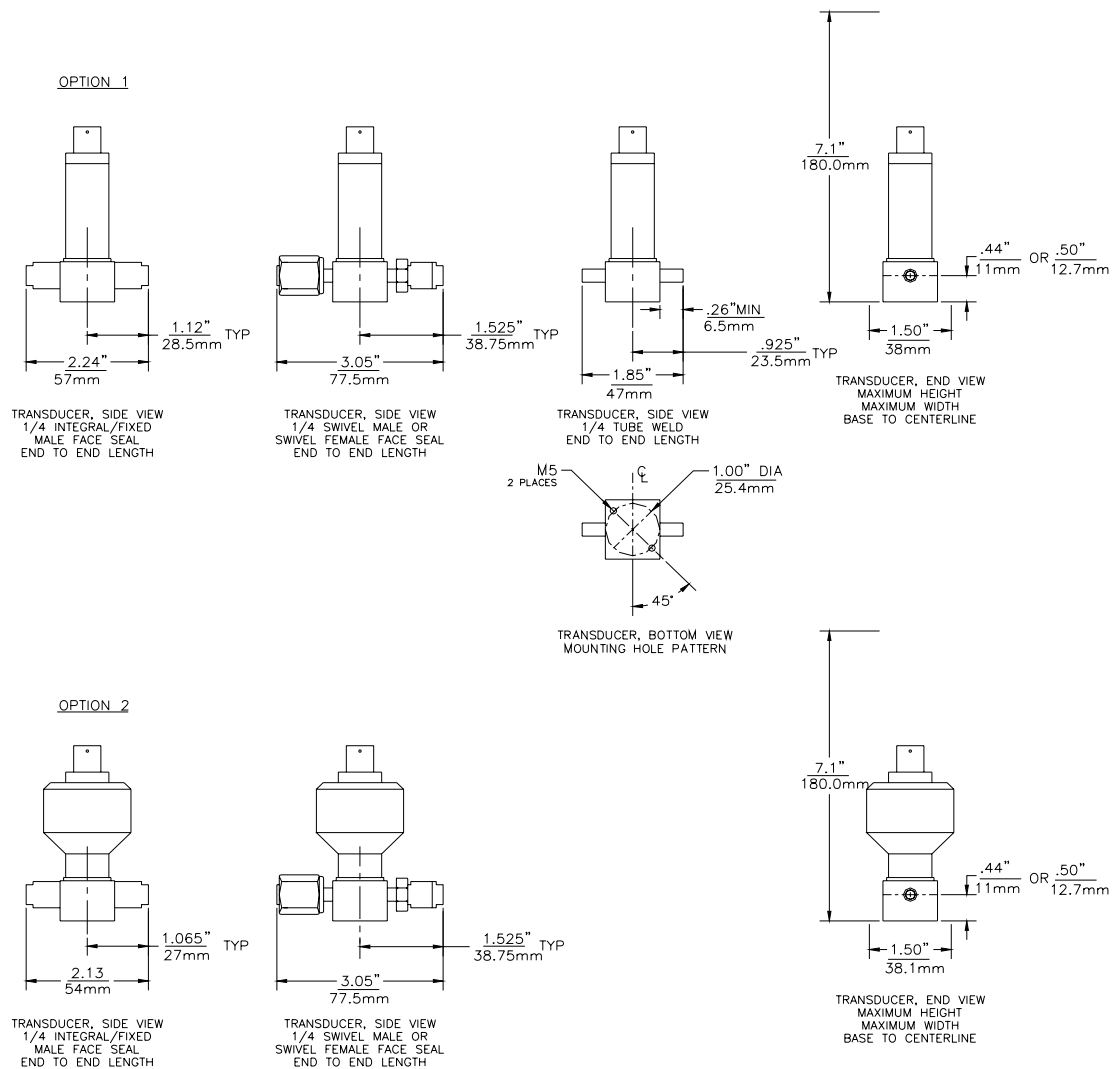


Figure 5
Pressure Transducer Interface Design

Table 5 Pressure Transducer Interface Recommendations

			Option 1 Pressure Transducers with Four-pin Connector		Option 2 Pressure Transducers with Six-pin Connector	
End-to-End Lengths: 1/4-inch Integral/fixed male face seal 1/4-inch Swivel face seal (male or female) 1/4-inch Tube weld			57 mm (2.24 in.) 77.5 mm (3.05 in.) 47 mm (1.85 in.)		54 mm (2.13 in.) 77.5 mm (3.05 in.) -	
NOTE 1: The minimum length for each tube weld stub should be 6.5 mm (0.26 in.). The tolerance on length for machined components is ± 0.15 mm (0.006 in.) from the component centerline to each end. The tolerance on length for welded components is ± 0.50 mm (0.020 in.) from the component centerline to each end.						
Overall Component Envelope: Maximum height Maximum width			180 mm (7.1 in.) 38 mm (1.5 in.)			
Base to Centerline of Flow Path:			11 mm (0.44 in.) ± 0.3 mm (0.012 in.) or 12.7 mm (0.50 in.) ± 0.3 mm (0.012 in.)			
Mounting Provisions: Threaded holes Pattern			M5 × 0.8 – 6H; 6 mm (0.24 in.) perfect thread depth 25.4 mm (1.00 in.) bolt circle centered on bottom of base with two threaded holes 180° apart and 45° off of the vertical or horizontal axis. One hole in each of the upper left and lower right quadrants when looking at the bottom of the component.		None required -	
NOTE 2: The M5 × 0.8 threaded mounting holes will accept 10–32 screws, but 10–32 threaded holes will not accept M5 × 0.8 screws.						
Fittings: Process			1/4-inch			
Control:						
Option 1 Electrical Four-pin round connector Signal pin out as follows:			Option 2 Electrical Six-pin round connector Signal pin out as follows:			
	Amperage Configuration	Voltage Configuration		Amperage Configuration	Voltage Configuration	
Pin A	+4–20 mA excitation	+ power input	Pin A	+4–20 mA excitation	+ power input	
Pin B	optional (open if not used)	pressure output/signal output	Pin B	optional (open if not used)	optional (open if not used)	
Pin C	optional (open if not used)	optional (open if not used)	Pin C	-4–20 mA negative	power and signal ground	
Pin D	-4–20 mA negative	power and signal ground	Pin D	optional (open if not used)	optional (open if not used)	
			Pin E	optional (open if not used)	pressure output/signal output	
			Pin F	optional (open if not used)	optional (open if not used)	



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SEMI F37-0299 (Reapproved 1104) METHOD FOR DETERMINATION OF SURFACE ROUGHNESS PARAMETERS FOR GAS DISTRIBUTION SYSTEM COMPONENTS

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 July 11, 2004. Initially available at www.semi.org September 2004; to be published November 2004. Originally published February 1999.

1 Purpose

1.1 The purpose of this test method is to define a method for determining numerical values for surface roughness parameters measured on gas distribution system components. Application of this method is intended to yield comparable data among users of this method.

2 Scope

2.1 This document will specify methods, measuring equipment, and test conditions for mechanical profile surface roughness measurement of gas distribution system components. This test method will not require nor recommend numerical values for specific surface roughness parameters. This test method is intended for use in quality control and process development for specification of and manufacturing of gas distribution system components.

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

3 Limitations

3.1 Numerical values for specific surface roughness parameters obtained using this test method may not provide sufficient information for determining the performance of the component, such as dry down times, corrosion resistance or amount of particle generation. Surface roughness measurement is one of many evaluation techniques for assessing the performance or quality of a surface. This document is not concerned with characteristics such as component design, material, dimensions, appearance, etc. While surface defects traversed by the stylus may be included in the roughness measurement, this method does not govern the evaluation of surface defects. Surface defects may be measured by methods such as SEM and EDX. This test method is intended to measure surface features on the order of 10 μm in size. It is not intended to detect submicron surface features.

4 Referenced Standards

4.1 ASME Standards¹

B46.1 — Surface Texture (Surface Roughness, Waviness, and Lay)

4.2 ASQ Standards²

Z1.9 — Sampling Procedures and Tables for Inspection by Variables for Percent Nonconforming

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *EDX* — Energy Dispersive X-Ray Spectroscopy

5.1.2 *SEM* — Scanning Electron Microscopy

5.1.3 *SEM* — Scanning Electron Microscope

5.2 Definitions

5.2.1 *cutoff* — a length selected to limit the spacing of surface irregularities. It separates a surface's roughness from its waviness.

5.2.2 *distinct regions* — visually unique areas of a surface defined by patterns of lay or differences in the appearance of surface roughness. In-line bores of different sizes or orientations and each leg of a shaped component should be considered distinct regions.

5.2.3 *evaluation length* — the actual length over which surface roughness is assessed.

5.2.4 *lay* — the general direction of orientation of surface features.

5.2.5 *skid* — a stylus probe support which acts as both a filter and a datum for probe movements.

¹ The American Society of Mechanical Engineers, Three Park Avenue, New York, NY 10016-5990, 800-843-2763, <http://www.asme.org/>

² American Society for Quality, 600 North Plankinton Avenue, Milwaukee, WI 53203, <http://www.asq.org/>

5.2.6 *skidless* — a type of instrument that does not use an external skid attached to the probe to act as a datum. Instead, it references a datum plane internal to the measurement equipment.

5.2.7 *stylus* — the object which mechanically probes the surface.

5.2.8 *surface roughness* — the finer irregularities of the surface texture, usually including those irregularities that result from the inherent action of the production process, for example traverse feed marks from cutting tools [ASME B46.1-1995].

5.2.9 *surface texture* — repetitive or random deviations from the nominal surface that forms the three-dimensional topography of the surface. Surface texture includes roughness, waviness, lay, and flaws [ASME B46.1-1995].

5.2.10 *waviness* — the more widely spaced component of surface texture. Waviness may result from such factors as machine or workpiece deflections, vibrations, chatter, heat treatment, or warping strains. Roughness may be considered as superimposed on a “wavy” surface.

6 Sampling

6.1 Acceptability criteria should be specified by the user and be developed in accordance with published sampling plans such as ASQ Z1.9, Sampling Procedures and Tables for Inspection by Variables for Percent Nonconforming.

7 Apparatus

7.1 The instrument should be a stylus type as defined by ASME B46.1, Section 2. Due to the limitations of skidded instruments (Type IV and V), a skidless instrument (Type I) is preferable.

7.2 The instrument should be calibrated using standard laboratory practices and manufacturer's recommendations with a roughness metrology standard calibrated by a certified testing laboratory and traceable to NIST³.

7.3 In order to resolve the features of the surface, a stylus with a conical tipped radius of 5 μm (200 μin) should be used.

8 Units of Measure

8.1 Surface roughness should be described by means of the Roughness Average, R_a , as defined per ASME B46.1.

8.2 Measurements should be specified in micro-meters (μm) or microinches (μin).

9 Preparation of Apparatus

9.1 The surface should be clean and free from loose debris before taking a measurement.

9.2 The instrument should be sufficiently isolated from vibrations, which will artificially increase roughness measurements.

9.3 Surfaces inaccessible to the stylus probe may be exposed by sectioning a sample(s). Samples should be sectioned to avoid damage to the surface to be measured and should be cleaned appropriately to remove sectioning debris.

10 Procedure

10.1 Select a single random location for measurement within each distinct region of interest on each component.

10.2 Measurements shall be taken perpendicular to the lay. If this is not practical, measurements may be taken in the direction of process gas flow through the component.

10.3 Instrument cutoff length should be set to 0.800 mm (0.030 in.).

10.4 The evaluation length, or measurement length, should be at least 3.81 mm (0.150 in.) where sufficient length is available.

11 Reporting

11.1 Average R_a is defined as the average of all R_a measurements taken over a population.

11.2 Maximum R_a is defined as the maximum of all R_a measurements taken over a population.

11.3 Both Average R_a and Maximum R_a should be reported for a given population. A population can be defined as:

- a set of single measurements on multiple components
- a set of multiple measurements on a single component
- a set of multiple measurements on multiple components

³ National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899-3460, (301) 975-6478, <http://www.nist.gov/>

12 Precision and Accuracy

12.1 Some variance in Roughness Average is to be expected on a single surface from measurement to measurement. No two measurements (as taken by different operators, on different instruments, or at different locations on the surface of a component) will contain the same data points. Because different instruments will acquire data points at different rates, and evaluation lengths may differ, a typical Roughness Average measurement can include hundreds to tens of thousands of data points. Reported measurements on the same surface can be expected to deviate up to 20%.

13 Related Documents

13.1 *ANSI Standard*⁴

Y14.36 — Surface Texture Symbols

13.2 *BSI Standard*⁵

BS 1134 — Assessment of Surface Texture, Part 1: Methods and Instrumentation and Part 2: Guidance and General Information

13.3 *DIN Standard*⁶

DIN 4768 — Determination of Surface Roughness Values R_a , R_z , R_{max} with Electric Stylus Instruments

13.4 *ISO Standard*⁷

ISO 4288 — Rules and Procedures for the Measurement of Surface Roughness Using Stylus Instruments

13.5 *JIS Standard*⁸

JIS B 0651 — Instruments for the Measurement of Surface Roughness by the Stylus Method

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4 American National Standards Institute, Headquarters: 1819 L Street, NW, Washington, DC 20036, USA. Telephone: 202.293.8020; Fax: 202.293.9287, New York Office: 11 West 42nd Street, New York, NY 10036, USA. Telephone: 212.642.4900; Fax: 212.398.0023, Website: www.ansi.org

5 British Standards Institution, 389 Chiswick High Road, London, W4 4AL, United Kingdom, +44 (0)20 8996 9000, <http://www.bsi-global.com/>

6 DIN Deutsche Institut für Normung, (DIN German Institut for standardization e.V.), Castle count road 6, 10787 Berlin, Germany, +49 30 2601-0, +49 30 2601-1231, <http://www2.din.de/>

7 International Standards Organization, 1, rue de Varembeé, Case postale 56, CH-1211 Geneva 20, Switzerland, +41 22 749 01 11; Fax: +41 22 733 34 30, <http://www.iso.org>

8 Japanese Standards Association, 4-1-24 Akasaka Minato-ku, Tokyo 107-8440 Japan, +81-3-3583-8005, Fax: +81-3-3586-2014, http://www.jsa.or.jp/default_english.asp

SEMI F38-0699 (Reapproved 1104)

TEST METHOD FOR EFFICIENCY QUALIFICATION OF POINT-OF-USE GAS FILTERS

This test method was technically reapproved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on August 16, 2004. Initially available on www.semi.org September 2004; to be published November 2004. Originally published June 1999.

1 Purpose

1.1 The purpose of this document is to define a comprehensive standard test sequence to qualify the particle filtration efficiency achievable using Point-of-Use (POU) gas filters.

2 Scope

2.1 This test method defines an evaluation method for Point-of-Use filters of various media (e.g., metallic, ceramic, and polymeric) typically used for filtering inert and process gases in semiconductor applications. Point-of-Use filters are designed to handle relatively low flow rates (0.5 – 50 slm.) and moderately high pressure drops. The filter housing and filtration element are combined into one sealed and inseparable unit.

2.2 This test method is intended to demonstrate the ability of a Point-of-Use gas filter to equal or exceed a specific particle filtration efficiency class when challenged with a monodispersed aerosol in the size range described in Section 6.6.

2.2.1 The efficiency class of the test method is defined as the log reduction value (LRV), where LRV is the Log [Input Concentration/System Background Level].

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

3 Limitations

3.1 These test methods do not apply to gas filter cartridges.

3.2 These test methods do not apply to bulk gas filters.

3.3 The various media (e.g., metallic, ceramic, and polymeric) may have slightly different critical particle sizes (most penetrating particle sizes). Therefore, many challenge tests have to be performed over a range of particle sizes to assure that the “worst case” challenge has been performed.

3.4 The efficiency test is for steady state flow and room temperature only. Comparison to efficiency in pulsed flow, elevated temperature etc., will have to be developed separately.

3.5 Statistical evaluation on the variability in efficiency of a media type should be done with a valid sample size. This evaluation is not within the scope of this test method.

3.6 The upper concentration limit of the CNC as specified by the manufacturer should not be exceeded. Exceeding the CNC concentration will understate the challenge concentration due to coincidence counting, thus affecting filter efficiency calculations.

3.7 This is not intended as a method for precise determination of filter efficiency. It is intended as a means for determining if a filter has an efficiency equal to or better than an approximate nominal value.

3.8 Commercially available radioactive neutralizers have pressure limitations. Users should be aware of these limits.

4 Referenced Standards

4.1 None.

5 Summary of Method

5.1 The test sequence consists of three steps:

5.1.1 Static background with filter.

5.1.2 Filter challenge with first monodispersed particle size (see Section 6.6 for particle size test sequence).

5.1.3 Filter challenge with subsequent monodispersed particle sizes.

6 Apparatus and Facility

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

6.2 *Particle Detector* — A condensation nucleus counter (CNC) with a counting efficiency of 50% at 0.01 micron, as reported by the manufacturer, is recommended to measure total particle concentration of particles greater than 0.01 micron. The CNC must meet

a background level as specified in Section 7.1.2.

6.3 Test Environment — Particle challenge testing does not require a clean area. Testing in a Class 100 area is recommended but not required. Refer to Section 6.7 for installation precautions.

6.4 Configuration for Efficiency Test — One example of the configuration is shown in Figure 1. The setup shown in Figure 1 is intended only as a guideline. This system consists of:

- an aerosol generation system,
- aerosol concentration detectors,
- an air flow measuring system,
- a filter pressure drop sensor, and
- the test filters.

6.5 Test Flow Rate — Maximum manufacturer's rated flow rate.

6.6 Challenge Particle Sizes — Experimentation of various media (e.g., metallic, ceramic, and polymeric) have resulted in most penetrating particle sizes ranging from 0.05–0.11 micron.^{1 2 3 4}

6.6.1 Several monodispersed particle challenge tests shall be done over a range of particle sizes so that testing at the most penetrating particle size is performed. An electrostatic classifier must be used to produce a monodispersed challenge.

6.6.2 Due to the effects of particle loading in relation to filtration efficiency, a clean filter should be used for each particle size challenge. The differential pressure across the test filter should be continuously monitored to eliminate the possibility of particle loading. The following particle increments are a guideline. Smaller increments are achievable and are desirable.

6.6.2.1 Challenge Particle Size In Microns:

1 Rubow, K.L., C.B. Davis (1991) "Particle Penetration Characteristics of Porous Metal Filter Media for High Purity Gas Filtration", Proceedings of Institute of Environmental Sciences Annual Technical Meeting

2 Rubow, K.L., and Liu, B.Y.H., Evaluation of Ultra-High Efficiency Membrane Filters, Proceedings of the 30th Annual Technical Meeting, Institute of Environmental Sciences, 1984.

3 Rubow, K.L., Liu, B.Y.H., and Grant, D.C., Characteristics of Ultra-High Efficiency Membrane Filters in Gas Applications; Journal of Environmental Sciences, May/June, 1988.

4 B. Gotlinsky, P. Conner, D. Capitanio, L. Johnson, and S. Tousi, Testing of All-Metal Filters for High Purity Semiconductor Process Gases, Proceedings of the 37th Annual Technical Meeting, Institute of Environmental Sciences, 1991.

- 0.05
- 0.07
- 0.10

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

6.8 Customary practices shall be employed for the design of the sampling system. This includes any specialized equipment or procedures recommended by the CNC manufacturer. Every effort should be taken to minimize differences in the particle concentrations of the sampling volume relative to the actual test volume. Care should be taken to provide an adequate exhaust length so that back diffusion of particles does not affect background at 2 times the sampling flow rate. This length will depend upon the particle concentration in the ambient environment and on other factors.

6.9 Test Particle — Solid non-volatile particles. Sodium chloride is recommended, but other particles could be used such as polystyrene latex beads (PSL) if sufficiently high concentrations can be generated.

7 Procedure

7.1 Static Background Test

7.1.1 Purge the system at a high velocity flush of 100 slm or the maximum flow rate that the system components will allow.

7.1.2 Purge the system at test flow rate (Section 6.5). Measure the particle concentration with clean gas to determine the background concentration. The background count is established when the counter has sampled a minimum of 3 scf (85 sl), and the arithmetic average during the last 3 scf (85 sl) of gas sampled is <2 particles/scf (<0.07 particles/sl). The sample interval shall be 1 minute in duration. Ensure that the background counts are stable or decreasing. If background cannot be achieved after 6 scf (170 sl) have been sampled, there may be a problem with the counter or test apparatus. Past experience has shown this to be an adequate volume of gas to give a satisfactory low level background particle counts.

7.2 Penetration Test