

SEMI F22-1102

GUIDE FOR GAS DISTRIBUTION SYSTEMS

This guide was technically approved by the Global Facilities Committee and is the direct responsibility of the North American Facilities Committee. Current edition approved by the North American Regional Standards Committee on July 21, 2002. Initially available at www.semi.org October 2002; to be published November 2002. Originally published June 1997.

1 Purpose

1.1 This reference document is intended to outline for the user the common systems configurations, components, and subcomponents of high purity gas distribution systems in a semiconductor fabrication facility. Related specifications are also noted.

2 Scope

2.1 Outlined in this document are both bulk and specialty gas distribution systems.

2.2 Components and subcomponents are identified from the point-of-supply to the point-of-connection to the process tool.

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

3 Limitations

3.1 The gas distribution system configurations noted in this reference document are provided solely as a guide to indicate common industry practice. They are not presented as, or intended to be, a representation of best engineering practice.

3.2 The reference documents noted are provided for general guidance only and are not intended to portray a code compliance protocol. The users should always consult local authorities to determine specific applicable codes and regulations. Agencies, regulatory or otherwise, noted herein are representative only of the U.S.

3.3 Specifications, SEMI Standards or other, are noted to assist the user as a reference only.

4 Referenced Standards

None.

5 Terminology

5.1 Acronyms and Abbreviations

5.1.1 *AOV* — Air-operated valves

5.1.2 *ASO* — Automatic shutoff valve

5.1.3 *POC* — Point of connection

5.1.4 *POU* — Point of use

5.1.5 *VMB* — Valve manifold box

5.2 Definitions

5.2.1 *air-operated valves (AOV)* — are those which require pneumatic energy to initiate or terminate flow or to change flow path (e.g., normally closed, partially open, double acting).

5.2.2 *automatic shutoff valve (ASO)* — a mechanically, electrically, or pneumatically activated valve which has the sole purpose of terminating flow if a predetermined condition is exceeded. For cryogenic supply systems, ASO's are used in conjunction with a low temperature sensing device.

5.2.3 *check valve* — a mechanical gas system component which prevents reverse flow.

5.2.4 *effluent treatment system* — a device which, through mechanical, chemical, cryogenic, absorbent, or other means, abates hazardous gas effluent to "environmentally safe levels" through dilution, neutralization, entrapment, or distillation.

5.2.5 *excess flow device* — a mechanical or electrical component which senses and signals an AOV or itself to terminate flow in the event predetermined flow is exceeded.

5.2.6 *face seal fitting* — a high purity fitting which incorporates two machined faces and a metallic gasket within a male/female nut configuration to attain a high leak integrity seal.

5.2.7 *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.

5.2.8 *flow restrictor* — a component, generally an orifice, which prohibits gas flow beyond a predetermined flow.

5.2.9 *gas box* — a gas distribution subsystem which generally contains the final shut-off valve(s) prior to the POC. It may also contain filter(s) and a regulator.

5.2.10 *hazardous gas detectors* — analytical instruments which placed in strategic locations in and

around gas distribution systems and components are used to detect potentially unnoticeable releases at extremely low levels.

5.2.11 *low temperature sensing device* — a component which protects the system downstream of the vaporizer from cryogenic temperatures by initiating an alarm or triggering a valve shut-down.

5.2.12 *nitrogen generation plant* — a system which, generally through cryogenic distillation, separates and purifies nitrogen from ambient air. Other air separation techniques may be used but are not considered in this document.

5.2.13 *point of connection (POC)* — is often used interchangeably with POU, indicating the final connection between the system and the process tool.

5.2.14 *point of use (POU)* — is often used interchangeably with POC, indicating the final connection between the system and the process tool.

5.2.15 *pressure relief valve* — a device which, at a given design pressure set point, releases gas pressure to prevent system over-pressurization.

5.2.16 *pressure transducer* — a component which mechanically or electrically senses the pressure within a gas system and transmits a signal to a readout or a control device.

5.2.17 *process gas panel* — a subsystem, generally contained within a gas cabinet, that delivers process gas from the cylinder to the specialty gas distribution system.

5.2.18 *purifier* — generally a catalytic, resinous, or diatomaceous material within a pressure vessel which removes particulate and/or trace gas impurities from a gas stream.

5.2.19 *regulator* — generally a mechanical device which alters the pressure within a gas system.

5.2.20 *segregation* — a practice in design and operation to prevent non-compatible gases from comingling.

5.2.21 *tube trailer* — a bulk gas supply system which manifolds high pressure, DOT-specified, vessels (cylindrical tubes) on a portable trailer.

5.2.22 *vacuum generator* — a component in a process gas panel which, via a suction created by an inert gas venturi, allows for evacuation of the gas system to levels of 16.7 kPa (25" mercury) or less.

5.2.23 *valve manifold box (VMB)* — a gas distribution subsystem designed to allow the distribution of a single process cylinder gas to multiple process tools. Its manifold design uses a series of valves (manual or

AOV), regulators, filters, and vacuum generator(s), and may be controlled manually or automatically.

5.2.24 *vaporizer* — a component which, through heat transfer, is designed to convert a cryogenic liquid to a gas.

6 Bulk Gas Distribution Systems

6.1 Cryogenic Liquid Supply (see Figure 1)

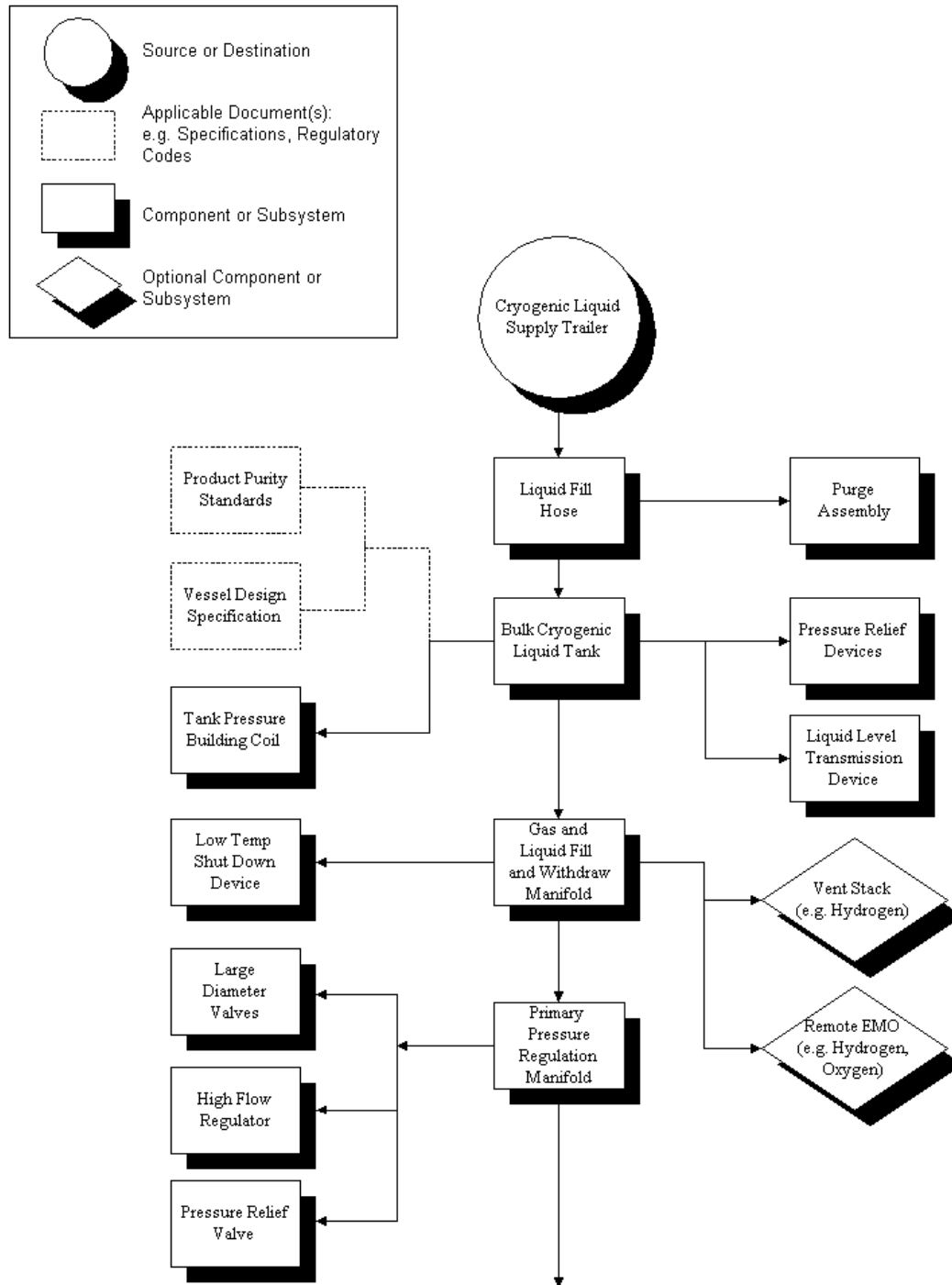


Figure 1
Bulk Gas (Cryogenic) Supply System Flow Chart

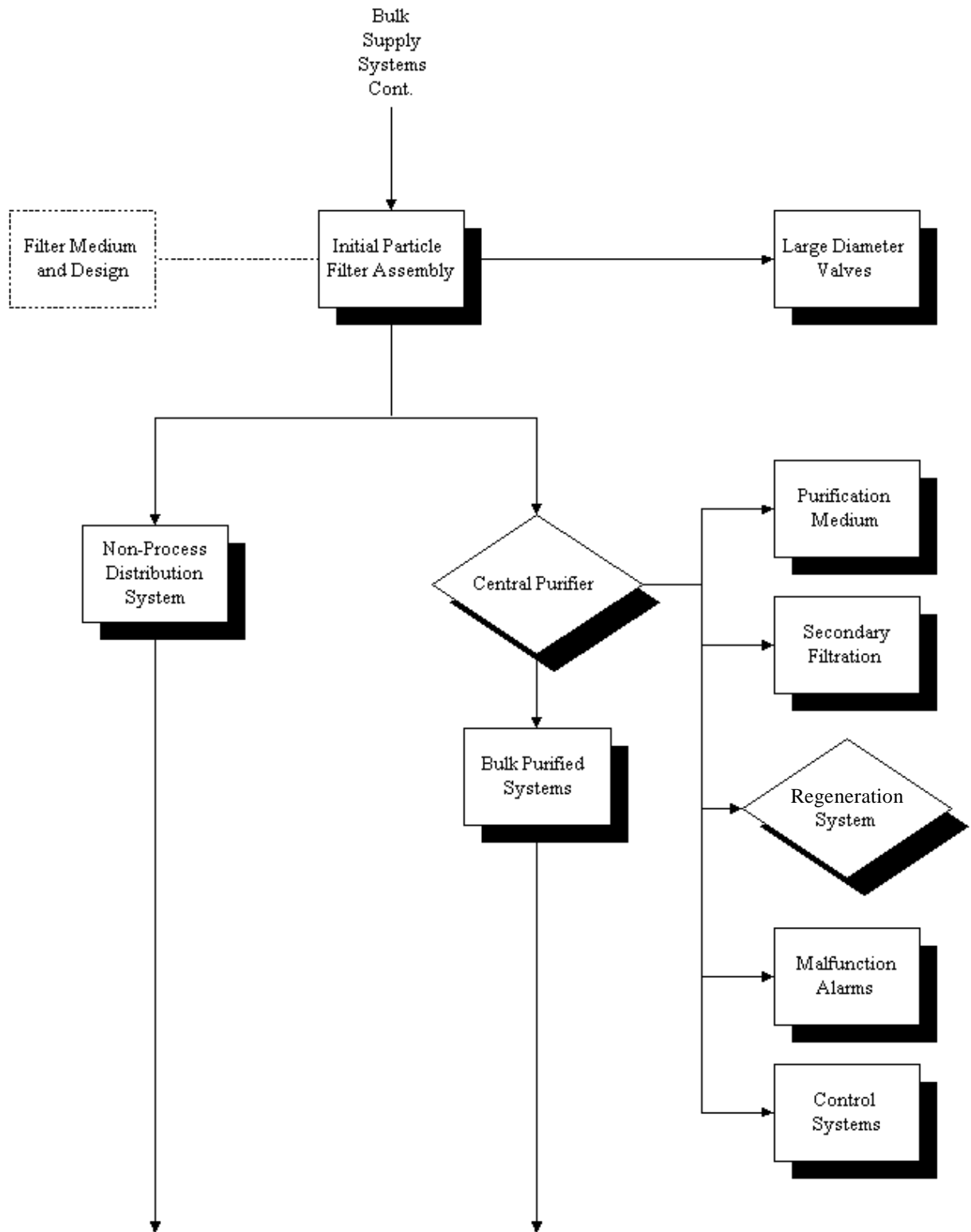


Figure 1
Bulk Gas (Cryogenic) Supply System Flow Chart (cont.)

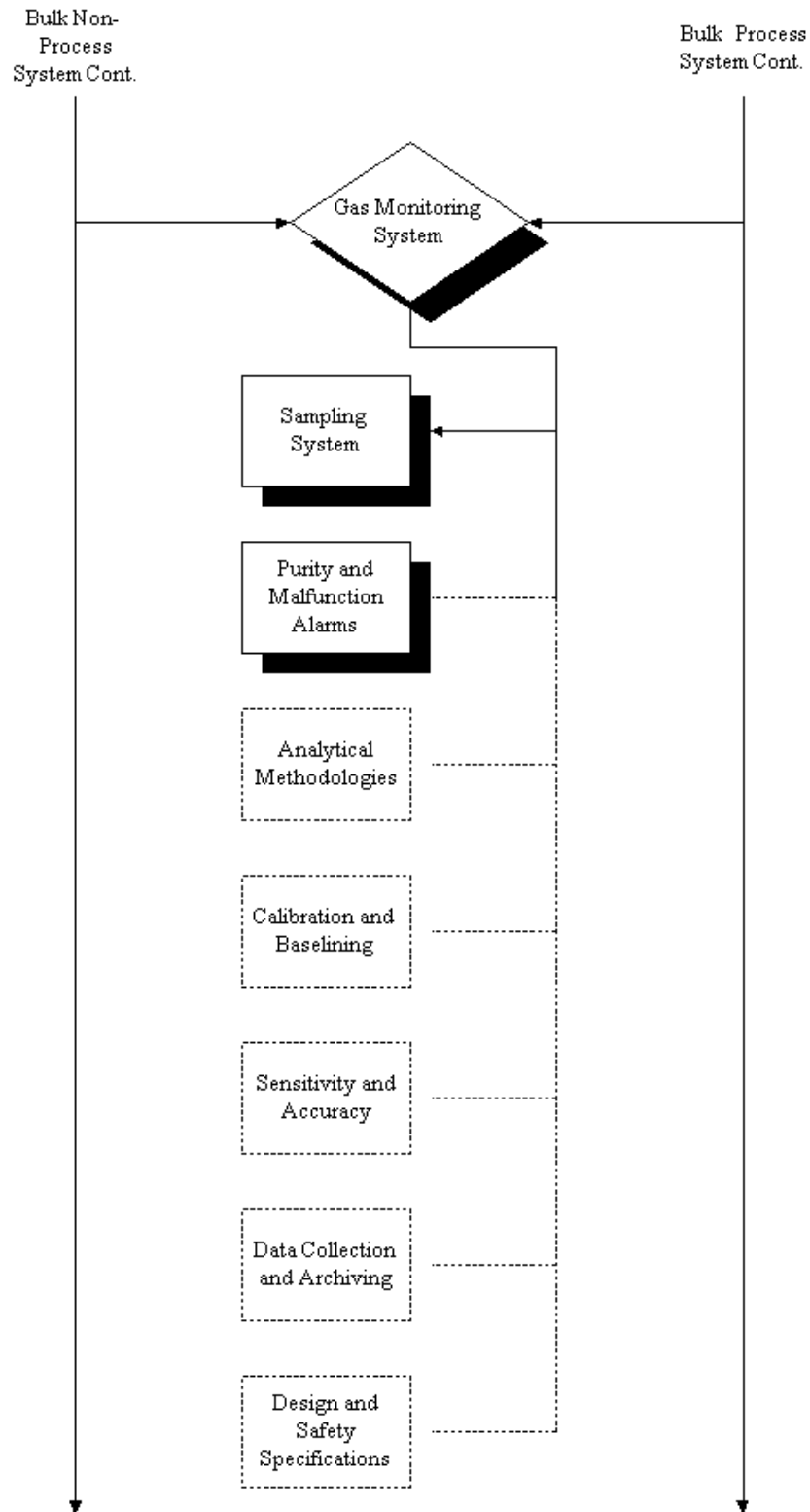


Figure 1
Bulk Gas (Cryogenic) Supply System Flow Chart (cont.)

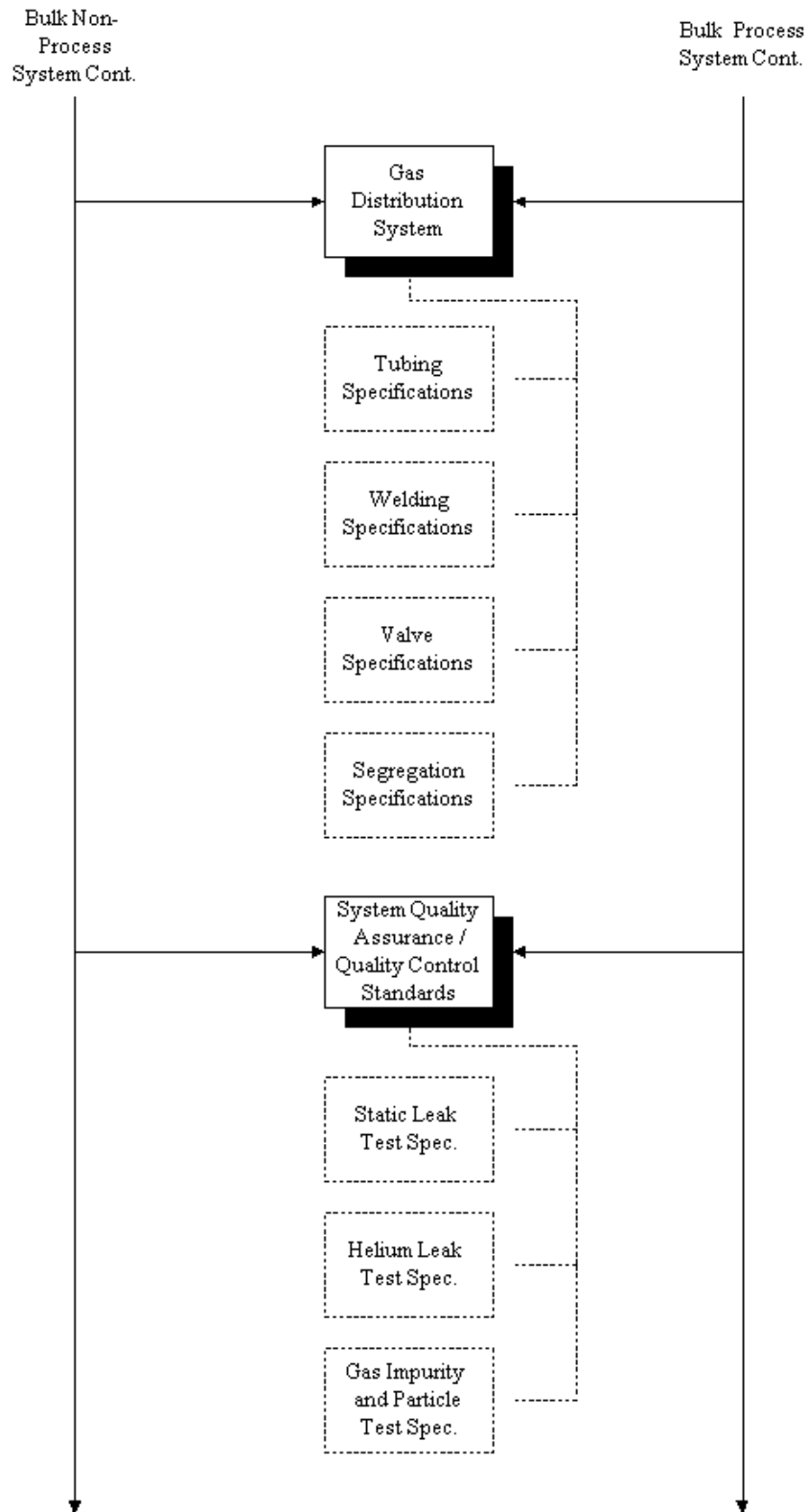


Figure 1
Bulk Gas (Cryogenic) Supply System Flow Chart (cont.)

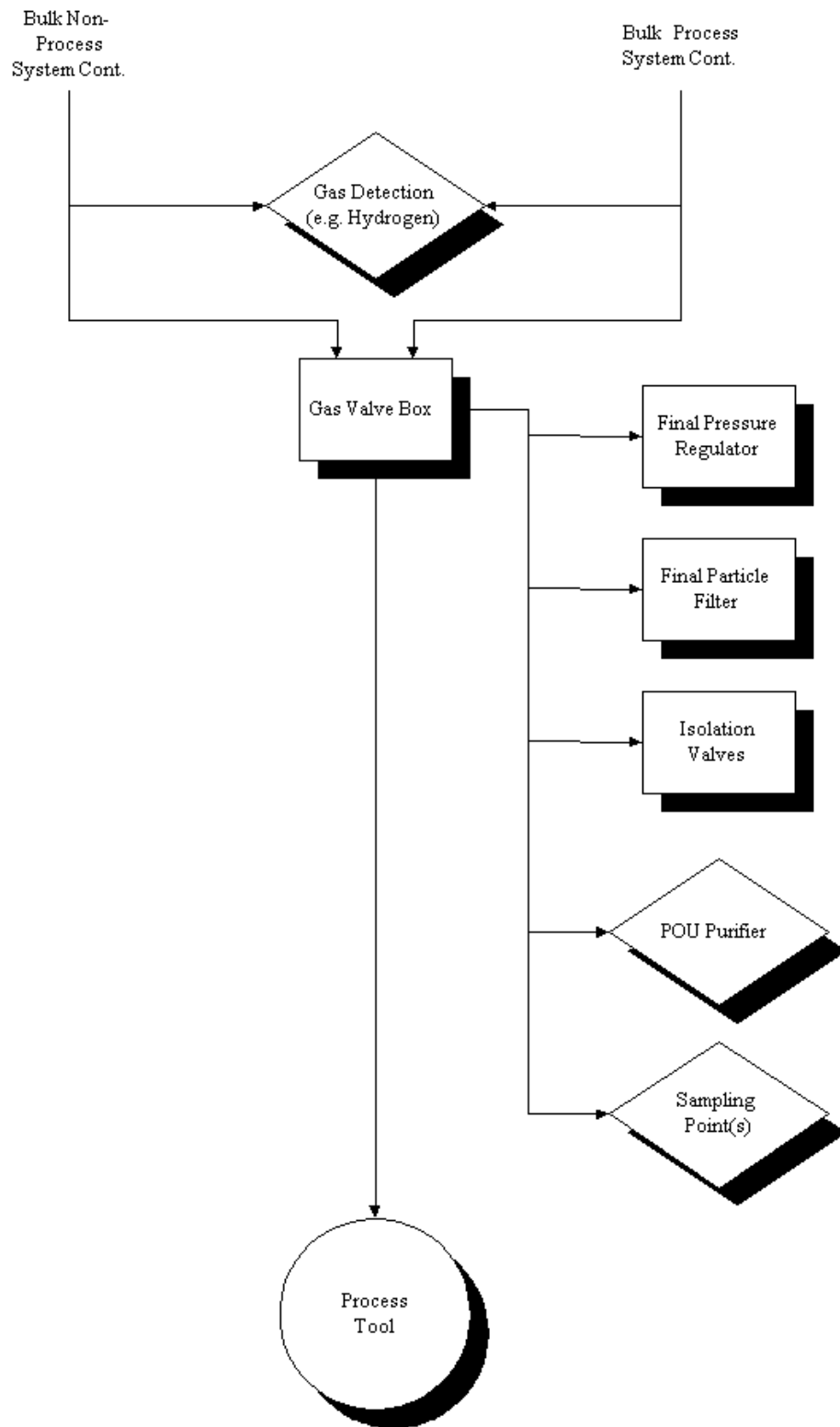


Figure 1
Bulk Gas (Cryogenic) Supply System Flow Chart (cont.)

- 6.1.1 *Cryogenic Liquid Storage Tank Pad*
 - 6.1.1.1 Product fill hose manifold
 - 6.1.1.2 Gas, liquid withdraw manifold
 - 6.1.1.3 Liquid vaporizers
 - 6.1.1.4 Pressure relief valve(s)
 - 6.1.1.5 Automatic shutoff valve (ASO)
 - 6.1.1.6 Low temperature sensing and shutoff device
 - 6.1.1.7 Liquid level indicator (local and remote)
 - 6.1.1.8 Vessel design specification (e.g., ASME¹ in US)
 - 6.1.1.9 Tank pressure building coil
 - 6.1.1.10 *Emergency Manual Off (EMO)* — hydrogen and oxygen only
 - 6.1.1.11 Product purity standards
- 6.1.2 *Primary Pressure Regulation Manifold*
 - 6.1.2.1 Regulator design specification
 - 6.1.2.2 Large diameter valve design specification
 - 6.1.2.3 Bypass configuration
 - 6.1.2.4 Pressure relief valves
- 6.1.3 *Initial Particle Filter Skid*
 - 6.1.3.1 Qualification standard
 - 6.1.3.2 Filtration medium design
 - 6.1.3.3 Bypass configuration
- 6.1.4 *Central Purifier*
 - 6.1.4.1 Purification medium, type, and qualification standards
 - 6.1.4.2 Regeneration requirements
 - 6.1.4.3 Electronics
 - 6.1.4.4 Control system
 - 6.1.4.5 Malfunction alarms
- 6.1.5 *Gas Monitoring System*
 - 6.1.5.1 Sampling system and standards
 - 6.1.5.2 Analytical methodologies and standards
 - 6.1.5.3 Calibration and baselining
 - 6.1.5.4 Sensitivity and accuracy
 - 6.1.5.5 Data collection and archiving

¹ American Society of Mechanical Engineers — An industry trade organization which sets standards for storage vessels, etc.

- 6.1.5.6 Purity standards and operational alarms
- 6.1.5.7 Test stand design and safety considerations
- 6.1.5.8 Portable system specifications
- 6.2 *High Pressure Tube Trailer Supply (See Figure 2)*
 - 6.2.1 Regional regulatory cylinder specification (e.g., DOT² in US region)
 - 6.2.2 Regional valve specification (e.g., CGA³ in US region)
 - 6.2.3 High pressure manifold
 - 6.2.4 Relief devices
 - 6.2.5 Excess flow devices (e.g., for H₂)
 - 6.2.6 Product purity standard
 - 6.2.7 Transfer hose
 - 6.2.8 Emergency manual off (EMO) (e.g., for H₂, O₂)
 - 6.2.9 Pressure billing gauge
- Tube retest specification (e.g., DOT in US region)
- 6.3 *On-Site Nitrogen Supply (see Figure 3)*
 - 6.3.1 Air compressor
 - 6.3.2 Production control system
 - 6.3.3 Air prepurifier for moisture and hydrocarbon removal
 - 6.3.4 Process instrumentation
 - 6.3.5 Control valves
 - 6.3.6 Distillation column
 - 6.3.7 Product booster compressor
 - 6.3.8 National electric codes
 - 6.3.9 Regional vessel guidelines (e.g., ASME (American Society of Mechanical Engineers) in US region)
 - 6.3.10 Vacuum-insulated piping for liquid add/withdraw
 - 6.3.11 EMO station
 - 6.3.12 Chiller

² Department of Transportation (DOT) — The US regulatory agency governing transport of hazardous materials.

³ Compressed Gas Association (CGA) — An industry trade organization which sets standards for cylinder outlet connections in the US.

7 Specialty Gas Distribution Systems

7.1 Process Gas Panel System (see Figure 4)

7.1.1 High Pressure

7.1.1.1 “Pigtail”/Cylinder connection assembly

7.1.1.2 Pressure transducer

7.1.1.3 Excess flow sensor

7.1.1.4 Nitrogen check valve

7.1.1.5 Nitrogen flow restrictor

7.1.1.6 Nitrogen purge valve

7.1.1.7 Vent valve

7.1.1.8 Process valve (regulator isolation)

7.1.1.9 Valve specifications

7.1.1.10 Tubing material specifications

7.1.2 Low Pressure

7.1.2.1 Pressure relief valve

7.1.2.2 Pressure transducer

7.1.2.3 Particle filter

7.1.2.4 Vent valve

7.1.2.5 Process valve

7.1.3 Pressure Regulator and Component Specifications

7.1.4 Vacuum Generator System

7.1.4.1 Venturi

7.1.4.2 Venturi supply valve

7.1.4.3 Supply check valve

7.1.4.4 Vacuum/pressure gauge

7.2 Nitrogen Purge Panel System

7.2.1 High Pressure

7.2.1.1 “Pigtail”/Cylinder connection assembly

7.2.1.2 Pressure gauge

7.2.1.3 Vent valve

7.2.1.4 Process valve (regulator isolation)

7.2.1.5 Pressure regulator

7.2.1.6 Low Pressure

7.2.1.7 Pressure gauge

7.2.1.8 Particle filter

7.2.1.9 Process valve

7.3 Distribution Lines — Gas Delivery System (Facilities)

7.3.1 Tubing specifications

7.3.2 Welding specifications

7.3.3 Valve specifications

7.3.4 System design specifications

7.3.5 Segregation specifications (cross contamination prevention)

7.3.6 System Quality Assurance/Quality Control standards

7.3.6.1 Static leak test and specification

7.3.6.2 He leak rate test and specification

7.3.6.3 Particle generation test and specification

7.3.6.4 Gaseous impurity test and specification

7.3.7 Hazardous gas detection requirements

7.4 Gas Box

7.4.1 Secondary pressure regulator

7.4.2 Secondary particle filtration

7.4.3 Isolation valves

7.4.4 Sampling point(s)

7.4.5 POU purification

7.5 Valve Manifold Box

7.5.1 Face seal connection

7.5.2 Isolation valve

7.5.3 Micro weld fitting

7.5.4 3/8 inch or 1/2 inch 316L SS tubing manifold

7.5.5 Micro weld fitting

7.5.6 1/4 inch 316L SS tubing stick line

7.5.7 Isolation valve

7.5.8 Pneumatic isolation valve

7.5.9 Face seal connection

7.5.10 Purge gas inlet micro weld fitting

7.5.11 Pressure regulator

7.5.12 Flow through pressure transducer or dead space pressure transducer or pressure gauge

7.5.13 Excess flow device

7.5.14 Vent gas outlet micro tee weld fitting

7.5.15 Face seal connection

7.5.16 Isolation valve

7.5.17 Tubing to tool

Termination inside the tool gas jungle area with an isolation valve as part of the tool.

7.6 *Tool Gas Jungle*

7.6.1 Process gas segregation

7.6.2 Manual isolation valve

7.6.3 AOV

7.6.4 MFC

7.6.5 Final particle filtration

7.6.6 Hazardous gas detection

7.6.7 Control systems

7.6.8 Electronics

8 **Related Documents**

8.1 *SEMI Documents*

SEMI E12 — Standard for Standard Pressure, Temperature, Density, and Flow Units Used in Mass Flow Meters and Mass Flow Controllers

SEMI E16 — Guideline for Determining and Describing Mass Flow Controller Leak Rates

SEMI E17 — Guideline for Mass Flow Controller Transient Characteristics Tests

SEMI E18 — Guideline for Temperature Specifications of the Mass Flow Controller

SEMI E27 — Standard for Mass Flow Controller and Mass Flow Meter Linearity

SEMI E28 — Guideline for Pressure Specifications of the Mass Flow Controller

SEMI E29 — Standard Terminology for the Calibration of Mass Flow Controllers and Mass Flow Meters

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

SEMI F2 — Specification for 316L Stainless Steel Tubing for General Purpose Semiconductor Manufacturing Applications

SEMI F3 — Guide for Welding Stainless Steel Tubing for Semiconductor Manufacturing Applications

SEMI F4 — Specification for Pneumatically Actuated Cylinder Valves

SEMI F5 — Guide for Gaseous Effluent Handling

SEMI F6 — Guide for Secondary Containment of Hazardous Gas Piping Systems

SEMI F13 — Guide for Gas Source Control Equipment

SEMI F14 — Guide for the Design of Gas Source Equipment Enclosures

SEMI F15 — Test Method for Enclosures Using Sulfur Hexafluoride Tracer Gas and Gas Chromatography

SEMI F16 — Specification for 316L Stainless Steel Tubing Which is to be Finished and Electropolished for High Purity Semiconductor Manufacturing Applications

SEMI F17 — Specification for High Purity Quality Electropolished 316L Stainless Steel Tubing, Component Tube Stubs, and Fittings Made from Tubing

SEMI F19 — Specification for the Finish of the Wetted Surfaces of Electropolished 316L Stainless Steel Components

SEMI F20 — Specification for 316L Stainless Steel Bar, Extruded Shapes, Plate, and Investment Castings for Components Used in the High Purity Semiconductor Manufacturing Applications

SEMI S1 — Safety Guideline for Equipment Safety Labels

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

SEMI S4 — Safety Guideline for the Segregation/Separation of Gas Cylinders Contained in Cabinets

SEMI S5 — Safety Guideline for Flow Limiting Devices

SEMI S6 — Safety Guideline for Ventilation

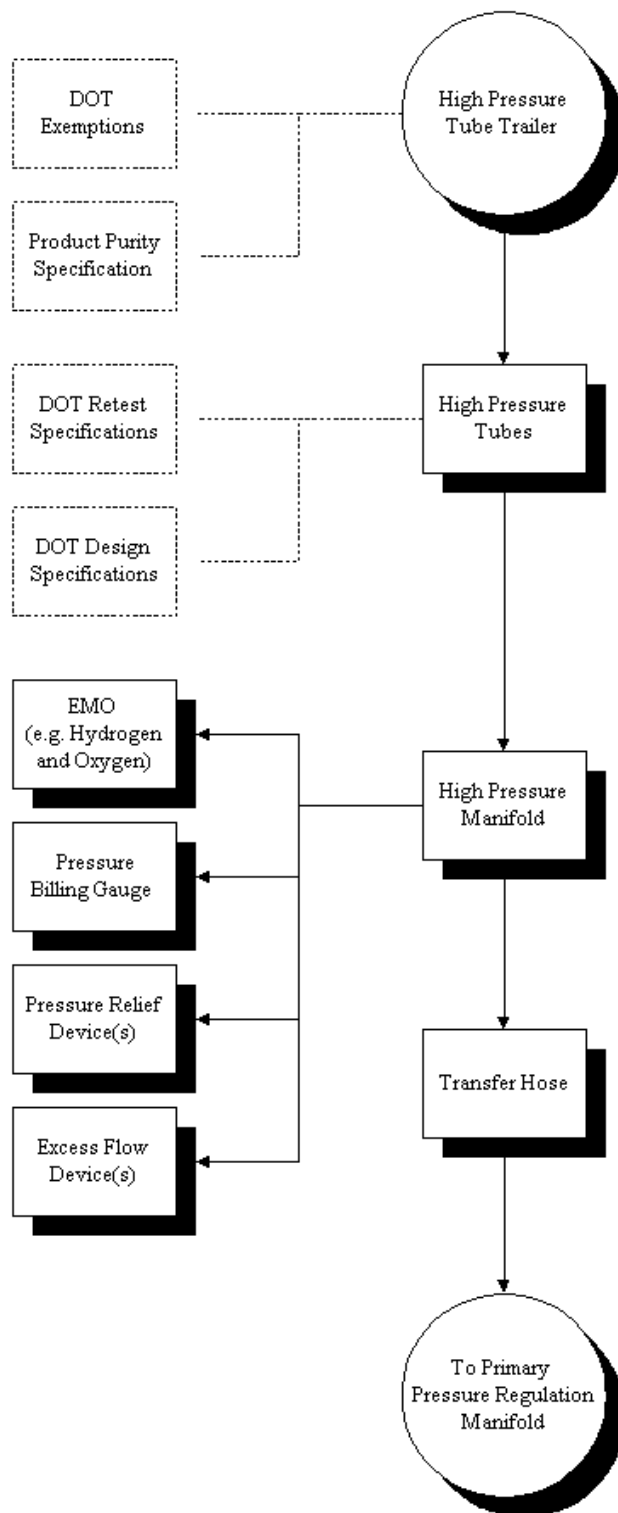


Figure 2
Bulk Gas High Pressure Gaseous Supply System Flow Chart

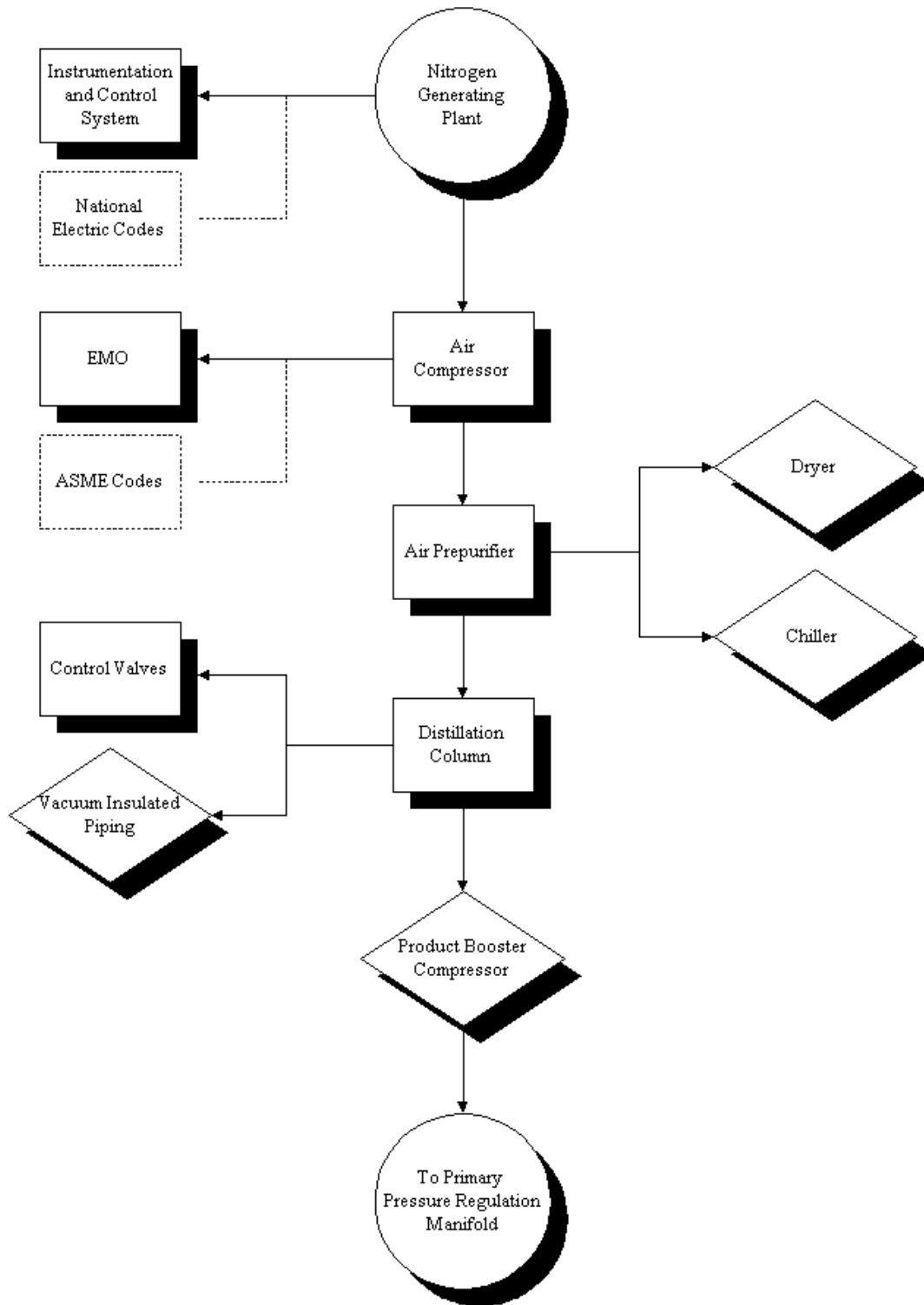


Figure 3
On-Site Nitrogen Supply System Flow Chart

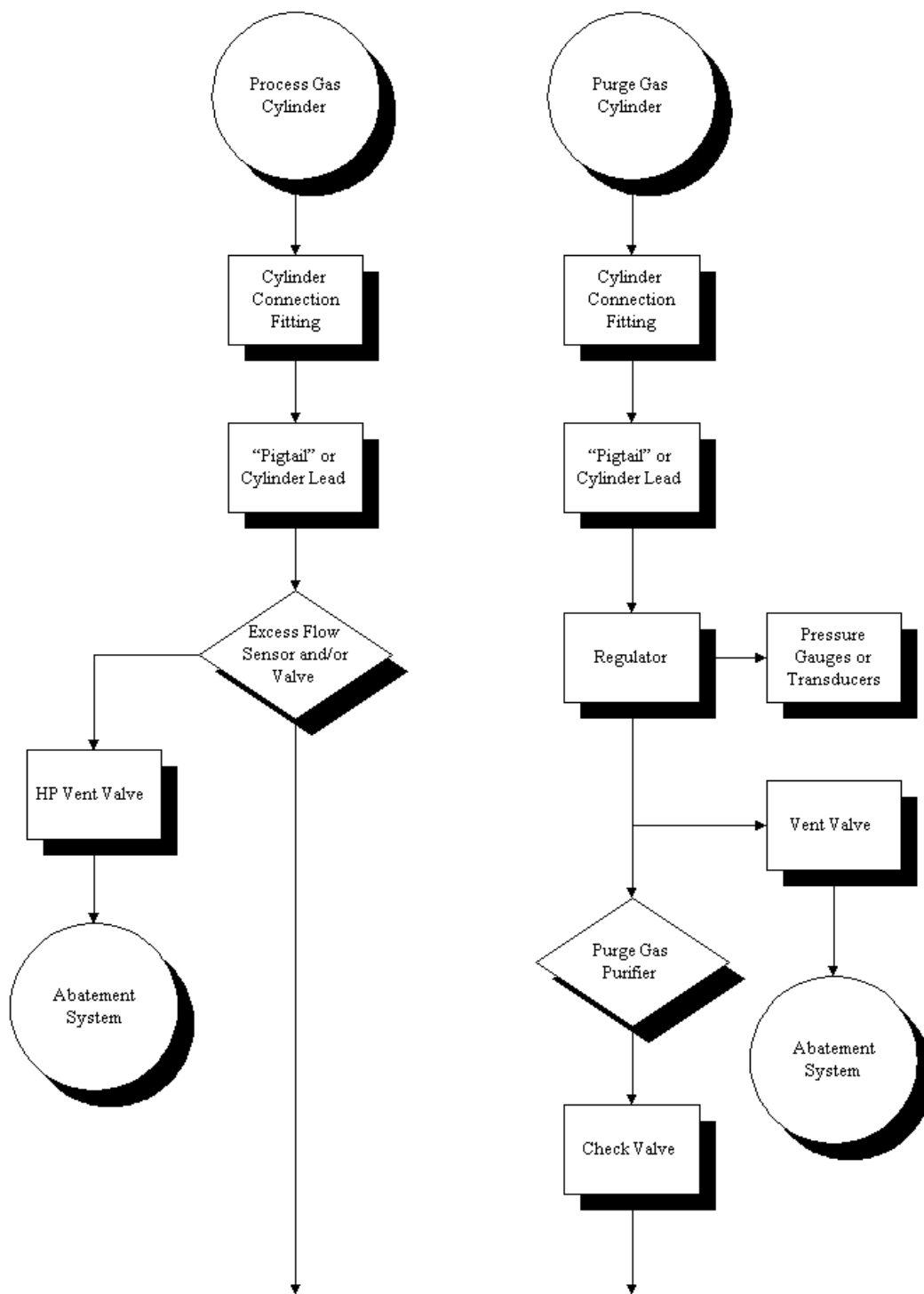


Figure 4
Specialty Gas Cabinet System Flow Chart

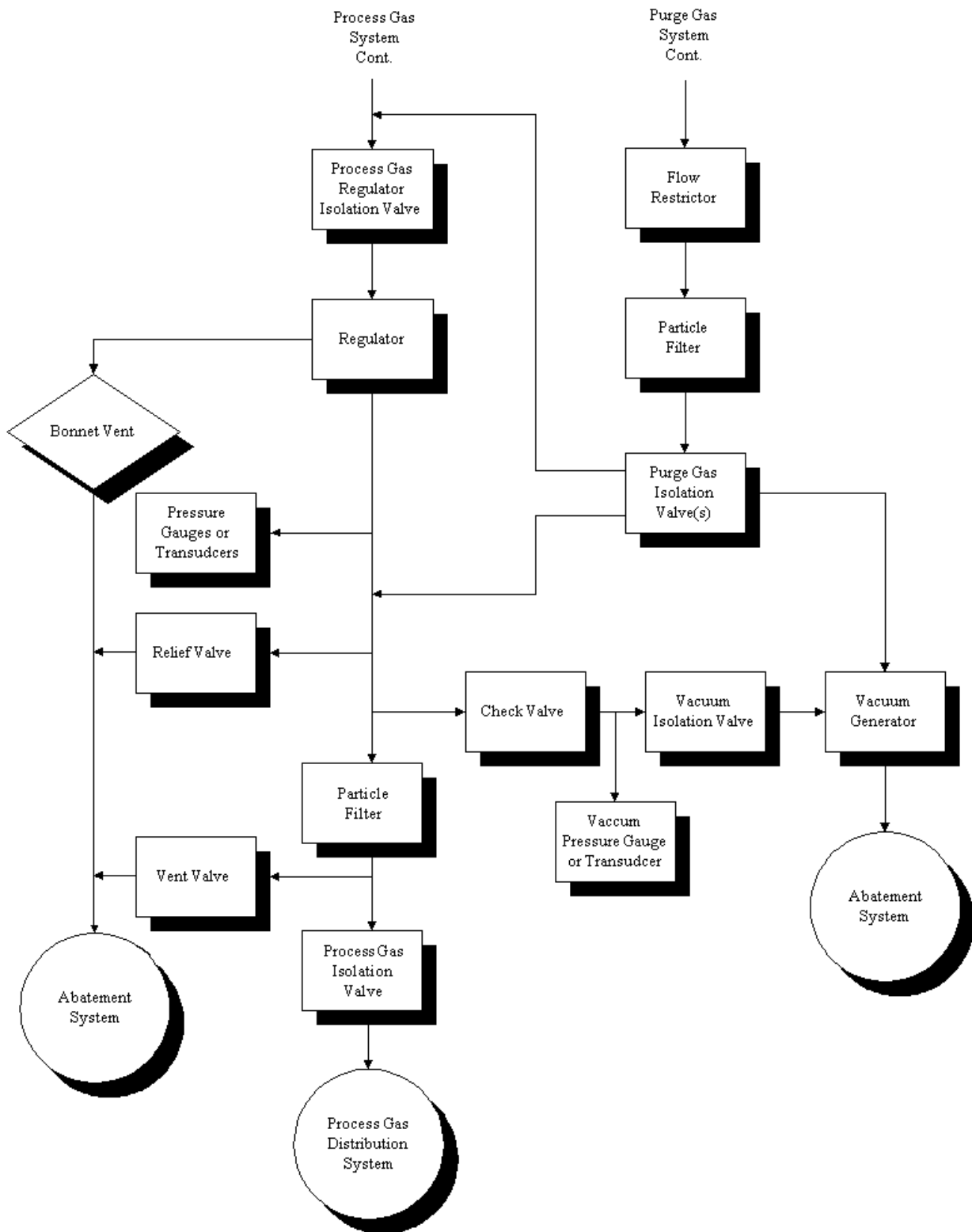


Figure 4
Specialty Gas Cabinet System Flow Chart (cont.)



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SEMI F23-0697 (Reapproved 0303)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 FLAMMABLE SPECIALTY GASES

This specification 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 October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published June 1997.

1 Purpose

1.1 The purpose of this document is to set a maximum permissible particle concentration for 10/0.2 grade flammable specialty gases and to describe a reference method for its verification.

2 Scope

2.1 This document applies only to flammable gases delivered through specialty gas systems at pressures up to 8×10^5 Pa (8 atmospheres). This method is not suitable for direct sampling from high pressure cylinders at pressures above 8×10^5 Pa (8 atmospheres). This document applies only to the following gas:

Hydrogen (H ₂)

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

3 Referenced Standards

3.1 SEMI Standard

SEMI C6.3 — Particle Specification for Grade 20/0.2 Hydrogen (H₂) Delivered as Pipeline Gas

3.2 JIS Standard¹

JIS B 9921 — Japanese Industrial Standard (1989), “Light Scattering Automatic Particle Counter”

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

4 Terminology

4.1 Variables

V_{Mi}	Volume of the i^{th} sample interval of the system gas
V_{Bi}	Volume of the i^{th} sample interval of the background

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

4.2 *gas sample volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1×10^5 Pa (1 atmosphere) pressure. Standard cubic feet (SCF) is defined at 21.1°C (70°F) and 1×10^5 Pa (1 atmosphere) pressure.

4.3 *average observed concentration of counts* (\bar{X}_M , \bar{X}_B) — The average concentration of counts, i.e.:

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

4.4 *calculated concentration of particles* (\bar{X}_C) — The concentration of particles in the system gas obtained by correcting the observed concentration in the system gas for the observed concentration in the background, i.e.:

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

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

¹ Japanese Industrial Standards, Available through the Japanese Standards Association, 1-24, Akasaka 4-Chome, Minato-ku, Tokyo 107-8440, Japan. Telephone: 81.3.3583.8005; Fax: 81.3.3586.2014 Website: <http://www.jsa.or.jp>

$$S_M = \sqrt{\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)}}$$

$$S_B = \sqrt{\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)}}$$

NOTE 1: The third is obtained from the first two, i.e.:

$$S_C = \sqrt{S_M^2 + S_B^2}$$

5 Requirements

5.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 6.

5.2 The specification will be considered met if the calculated concentration of particles plus 2 times the standard deviation does not exceed 10 particles per 25 standard liters, i.e.:

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

6 Apparatus

6.1 *Particle Counter* — An instrument suitable for counting particles in compressed flammable gases at a pressure of up to 8×10^5 Pa (8 atmospheres) with a 50% counting efficiency should reach 90% at 0.3 micrometer or smaller. The resolving power of the instrument near 0.2 micrometer should be no worse than 10%. The counting efficiency is determined by a calibration at 1×10^5 Pa (1 atmosphere) pressure using polystyrene latex spheres in an inert gas and a reference particle counter with a proven counting efficiency of not less than 95% at 0.2 micrometer.

NOTE 2: Suitable test methods for determining counting efficiency and resolving power are contained in Japanese Industrial Standard JIS B 9921 (1989), "Light Scattering Automatic Particle Counter". More sensitive particle counters result in a higher measured particle concentration.

7 Test Method

NOTE 3: The details of the sampling configuration, measurement procedure, and instrument calibration procedure and frequency, must be agreed upon by the user and supplier, taking into account good engineering practice. Material safety data sheets should be referred to for safe handling of specialty gases.

7.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing nitrogen, believed to be free of particles 0.2 micrometer or more in diameter, through the instrument and recording the total number of counts. The nitrogen purge assembly

for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Count a minimum of 3 equal intervals, each of at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Calculate \bar{X}_B as defined in Section 4. \bar{X}_B must not exceed 2 counts per 25 standard liters.

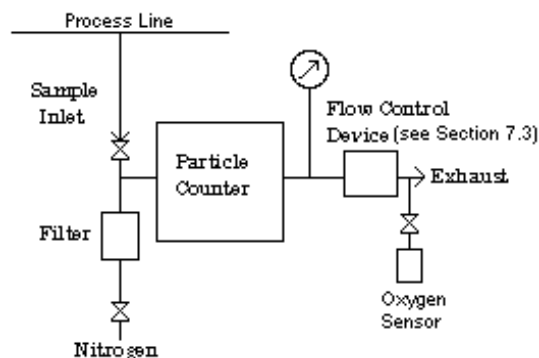


Figure 1
Schematic Diagram of Configuration for Obtaining Particle Samples from Flammable Specialty Gas Systems

7.2 The sampling point should be near the point of use, and sampling lines should be clean and as short as possible.

7.3 The sampling system configuration is shown in Figure 1. The specialty gas system should be connected through a sample valve directly to the particle counter, a pressure gauge, and a flow control device (FCD). When sampling is performed using wall tap sample ports, the aspiration efficiency should be checked using the method described in Related Information 1. The sampling system should contain minimum dead volume and sample tube length. All components in the sampling system should be leak tight and cleaned in accordance with good engineering practice. A leak check of the system should be performed in accordance with normally accepted practice. The FCD may be part of the particle counter. The particle counter should be maintained at the pressure of the gas system. The FCD can be a metering valve and a flow meter or a critical orifice. The exhaust line should be leak tight and piped to an appropriate exhaust system. The exhaust line diameter should be large enough to produce no more than 1×10^4 Pa (1.5 psi) pressure drop during purge and sample flow.

NOTE 4: The alternative sampling configurations described in SEMI C6.3 can also be used for hydrogen.

7.4 Using the high purity nitrogen system shown in Figure 1, purge the sampling system and particle counter for at least 5 minutes at the instrument manufacturer's specified flow rate. The exact purge time should take into account the reactivity of the gas

and should be sufficient to purge the entire exhaust line. An oxygen sensor should be placed at the system exhaust to verify less than 1 ppmv oxygen in the purge nitrogen. The purging must be performed before flammable gas is introduced into the sampling system and after completion of the measurement. The valve leading to the oxygen sensor should be closed when flammable gas is being sampled. The valve leading from the purge nitrogen system should be closed when sampling specialty gases and/or a back flow prevention device should be included in the purge nitrogen system.

NOTE 5: It is known that flammable gases can react with oxygen present in the sampling system. Nitrogen purging of the sampling system reduces the risk of reaction. The purity of the purge nitrogen should be sufficient to remove reactive substances from the sampling system.

7.5 Count the particles in each of at least 3 equal intervals. Each sample interval must be at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Data obtained during the first 5 minutes after the sample valve is opened may be discarded. Record the number of counts in the sample volume for each interval. Calculate \bar{X}_C and S_C , as defined in Section 4.

NOTE 6: For small volume specialty gas systems, the user and supplier may agree to a smaller sample gas volume than that stated above. The sample point location and the process line pressure and flow rate during the test should be recorded.

8 Report

8.1 The report shall contain the values of all the variables defined in Section 4.

9 Related Documents

Hart, J. J., W. T. McDermott, A. E. Holmer, and J. P. Natwora, Jr. Particle Measurement in Specialty Gases. *Solid State Technol.*, 38(9):111-116, September 1995.

Wang, H. C. and R. Udischas. Counting Particles in High Pressure Electronic Specialty Gases. *Solid State Technol.*, 37(6):97-107, June 1994.

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RELATED INFORMATION 1

NOTES ON ASPIRATION EFFICIENCIES OF WALL TAP SAMPLE PORTS

NOTICE: This related information is not an official part of SEMI F23 and is not intended to modify or supercede the official standard. Determination of the suitability of the material is solely the responsibility of the user.

R1-1 Introduction

R1-1.1 Isokinetic sample probes are usually not provided in speciality gas systems. Particle sampling is usually performed using wall tap sample ports. Wall tap sample ports are oriented 90° to the process line flow as shown in Figure R1-1. Non-isokinetic flow into the sample tube results from the 90° change in flow direction. Additional deviation from isokinesis can result from stream tube contraction or expansion when the velocities U and v are not equal. The effects of non-isokinetic flow on the measurement should be checked. The particle sampling process should be performed with an aspiration efficiency close to 1.

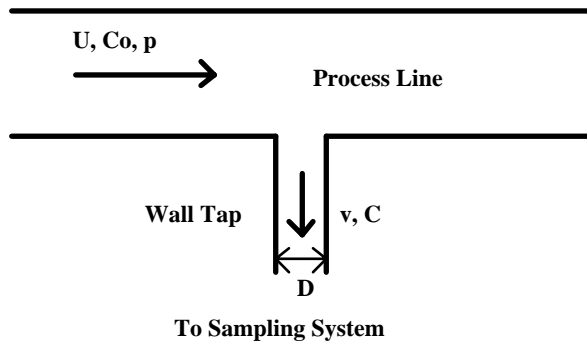


Figure R1-1

Schematic Diagram of Process Line and Wall Tap Sample Port

R1-2 Variables

d_p	Diameter of particle (cm)
ρ_p	Intrinsic density of particle (g/cm^3)
η	Dynamic viscosity of the gas (g/s-cm)
λ_o	Mean free path of the gas at atmospheric pressure (cm)
p	Pressure of the gas (Pa)
P_o	Atmospheric pressure ($= 1 \times 10^5$ Pa)
D	Diameter of wall tap sample tube (cm)
U	Average velocity of the process line flow (cm/s)
v	Average velocity of the sample line flow (cm/s)
C^*	Stokes-Cunningham slip correction factor
S_{tk}	Stokes number
R	Velocity ratio
Co	Particle concentration in the process line (cm^{-3})
C	Particle concentration in the sample line (cm^{-3})

R1-3 Calculations

R1-3.1 The Stokes-Cunningham slip correction factor is calculated for particles in the size range of interest:

$$C^* = 1 + 2.492 \frac{p_o \lambda_o}{p d_p} + 0.84 \frac{p_o \lambda_o}{p d_p} e^{-0.435 p d_p / p_o \lambda_o}$$

Calculate the velocity ratio and Stokes number. The intrinsic particle density may assume a worst case value of 10 g/cm^3 :

$$R = U/v$$

$$S_{tk} = \frac{d_p^2 \rho_p U C^*}{18 \eta D}$$

A wall tap sample port can be approximated as a sampling probe oriented 90° to the flow. Calculate the aspiration efficiency:

$$\frac{C}{Co} = \frac{1}{1 + 8 S_{tk} R^{1/2}}$$

Repeat the calculations for particle sizes in the range of interest. When the aspiration efficiency is significantly different from 1, the calculated efficiency should be used to correct the measured particle concentration, C .

R1-4 References

Stevens, D. C. Review of Aspiration Coefficients of Thin-Walled Sampling Nozzles. J. Aerosol Sci., 17(4):729-743, 1986.

Vincent, J. H., D. C. Stevens, D. Mark, M. Marshall, and T. A. Smith. On the Aspiration Characteristics of Large-Diameter, Thin-Walled Aerosol Sampling Probes at Yaw Orientations With Respect to the Wind. J. Aerosol Sci., 17(2):211-224, 1986.

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SEMI F24-0697 (Reapproved 0303)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 INERT SPECIALTY GASES

This specification 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 October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published June 1997.

1 Purpose

1.1 The purpose of this document is to set a maximum permissible particle concentration for 10/0.2 grade inert specialty gases and to describe a reference method for its verification.

2 Scope

2.1 This document applies only to inert gases delivered through specialty gas systems at pressures up to 8×10^5 Pa (8 atmospheres). This method is not suitable for direct sampling from high pressure cylinders at pressures above 8×10^5 Pa (8 atmospheres). This document applies only to the following gases:

Argon (Ar)
Halocarbon 23 (CHF ₃)
Halocarbon 116 (C ₂ F ₆)
Helium (He)
Nitrogen (N ₂)
Sulfur Hexafluoride (SF ₆)
Tetrafluoromethane (CF ₄)

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

3 Referenced Standards

3.1 SEMI Standard

SEMI C6.5 — Particle Specification for Grade 10/0.2 Nitrogen (N₂) and Argon (Ar) Delivered as Pipeline Gas

3.2 JIS Standard¹

JIS B 9921 — Japanese Industrial Standard (1989), “Light Scattering Automatic Particle Counter”

¹ Japanese Industrial Standards, Available through the Japanese Standards Association, 1-24, Akasaka 4-Chome, Minato-ku, Tokyo 107-8440, Japan. Telephone: 81.3.3583.8005; Fax: 81.3.3586.2014 Website: <http://www.jsa.or.jp>

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

4 Terminology

4.1 Variables

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

4.2 *gas sample volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1×10^5 Pa (1 atmosphere) pressure. Standard cubic feet (SCF) is defined at 21.1°C (70°F) and 1×10^5 Pa (1 atmosphere) pressure.

4.3 *average observed concentration of counts* (\bar{X}_M , \bar{X}_B) — The average concentration of counts, i.e.:

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

4.4 *calculated concentration of particles* (\bar{X}_C) — The concentration of particles in the system gas obtained by correcting the observed concentration in the system gas for the observed concentration in the background, i.e.:

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

4.5 *standard deviation* (S_M , S_B , S_C) — A statistical measure of the spread of the concentration of the counts or particles. The first two are obtained from the interval

and average concentrations and the number of intervals, i.e.:

$$S_M = \sqrt{\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)}}$$

$$S_B = \sqrt{\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)}}$$

NOTE 1: The third is obtained from the first two, i.e.:

$$S_C = \sqrt{S_M^2 + S_B^2}$$

5 Requirements

5.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 6.

5.2 The specification will be considered met if the calculated concentration of particles plus 2 times the standard deviation does not exceed 10 particles per 25 standard liters, i.e.:

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

6 Apparatus

6.1 *Particle Counter* — An instrument suitable for counting particles in compressed inert gases at a pressure of up to 8×10^5 Pa (8 atmospheres) with a 50% counting efficiency at 0.2 micrometer or smaller. The counting efficiency should reach 90% at 0.3 micrometer or smaller. The resolving power of the instrument near 0.2 micrometer should be no worse than 10%. The counting efficiency is determined by a calibration at 1×10^5 Pa (1 atmosphere) pressure using polystyrene latex spheres in an inert gas and a reference particle counter with a proven counting efficiency of not less than 95% at 0.2 micrometer.

NOTE 2: Suitable test methods for determining counting efficiency and resolving power are contained in Japanese Industrial Standard JIS B 9921 (1989), "Light Scattering Automatic Particle Counter". More sensitive particle counters result in a higher measured particle concentration.

7 Test Method

NOTE 3: The details of the sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice. Material safety data sheets should be referred to for safe handling of specialty gases.

7.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing nitrogen, believed to be free of particles 0.2 micrometer or more

in diameter, through the instrument and recording the total number of counts. The nitrogen purge assembly for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Count a minimum of 3 equal intervals, each of at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Calculate \bar{X}_B as defined in Section 4. \bar{X}_B must not exceed 2 counts per 25 standard liters.

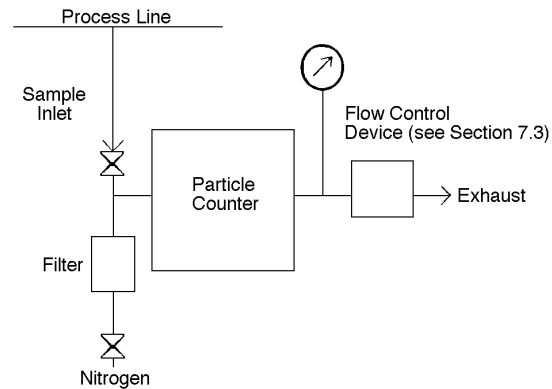


Figure 1
Schematic Diagram of Configuration for
Obtaining Particle Samples from Inert Specialty
Gas Systems

7.2 The sampling point should be near the point of use, and sampling lines should be clean and as short as possible.

7.3 The sampling system configuration is shown in Figure 1. The specialty gas system should be connected through a sample valve directly to the particle counter, a pressure gauge, and a flow control device (FCD). When sampling is performed using wall tap sample ports, the aspiration efficiency should be checked using the method described in Related Information 1. The sampling system should contain minimum dead volume and sample tube length. All components in the sampling system should be leak tight and cleaned in accordance with good engineering practice. A leak check of the system should be performed in accordance with normally accepted practice. The FCD may be part of the particle counter. The particle counter should be maintained at the pressure of the gas system. The FCD can be a metering valve and a flow meter or a critical orifice. The exhaust line should be leak tight and piped to an appropriate exhaust system. The exhaust line diameter should be large enough to produce no more than 1×10^4 Pa (1.5 psi) pressure drop during purge and sample flow.

NOTE 4: The alternative sampling configurations described in SEMI C6.5 can also be used for nitrogen or argon.

7.4 Using the high purity nitrogen system shown in Figure 1, purge the sampling system and particle

counter for at least 5 minutes at the instrument manufacturer's specified flow rate. The exact purge time should be sufficient to purge the entire exhaust line. The purging must be performed before sample gas is introduced into the sampling system and after completion of the measurement. The valve leading from the purge nitrogen system should be closed when sampling specialty gases and/or a back flow prevention device should be included in the purge nitrogen system.

7.5 Count the particles in each of at least 3 equal intervals. Each sample interval must be at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Data obtained during the first 5 minutes after the sample valve is opened may be discarded. Record the number of counts in the sample volume for each interval. Calculate \bar{X}_C and S_C , as defined in Section 4.

NOTE 5: For small volume specialty gas systems, the user and supplier may agree to a smaller sample gas volume than that stated above. The sample point location and the process line pressure and flow rate during the test should be recorded.

8 Report

8.1 The report shall contain the values of all the variables defined in Section 4.

9 Related Documents

Hart, J. J., W. T. McDermott, A. E. Holmer, and J. P. Natwora, Jr. Particle Measurement in Specialty Gases. Solid State Technol., 38(9):111–116, September 1995.

Wang, H. C. and R. Udischas. Counting Particles in High Pressure Electronic Specialty Gases. Solid State Technol., 37(6):97–107, June 1994.

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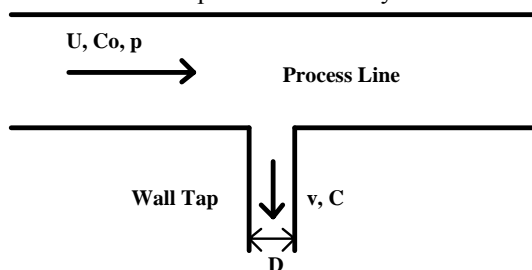
RELATED INFORMATION 1

NOTES ON ASPIRATION EFFICIENCIES OF WALL TAP SAMPLE PORTS

NOTICE: This related information is not an official part of SEMI F24 and is not intended to modify or supercede the official standard. Determination of the suitability of the material is solely the responsibility of the user.

R1-1 Introduction

R1-1.1 Isokinetic sample probes are usually not provided in speciality gas systems. Particle sampling is usually performed using wall tap sample ports. Wall tap sample ports are oriented 90° to the process line flow as shown in Figure R1-1. Non-isokinetic flow into the sample tube results from the 90° change in flow direction. Additional deviation from isokinesis can result from stream tube contraction or expansion when the velocities U and v are not equal. The effects of non-isokinetic flow on the measurement should be checked. The particle sampling process should be performed with an aspiration efficiency close to 1.



To Sampling System
Figure R1-1

Schematic Diagram of Process Line and Wall Tap Sample Port

R1-2 Variables

d_p =	Diameter of particle (cm)
ρ_p =	Intrinsic density of particle (g/cm ³)
η =	Dynamic viscosity of the gas (g/s-cm)
λ_o =	Mean free path of the gas at atmospheric pressure (cm)
p =	Pressure of the gas (Pa)
P_o =	Atmospheric pressure (= 1×10^5 Pa)
D =	Diameter of wall tap sample tube (cm)
U =	Average velocity of the process line flow (cm/s)
v =	Average velocity of the sample line flow (cm/s)
C^* =	Stokes-Cunningham slip correction factor
S_{tk} =	Stokes number
R =	Velocity ratio
Co =	Particle concentration in the process line (cm ⁻³)
C =	Particle concentration in the sample line (cm ⁻³)

R1-3 Calculations

R1-3.1 The Stokes-Cunningham slip correction factor is calculated for particles in the size range of interest:

$$C^* = 1 + 2.492 \frac{p_o \lambda_o}{p d_p} + 0.84 \frac{p_o \lambda_o}{p d_p} e^{-0.435 p d_p / p_o \lambda}$$

Calculate the velocity ratio and Stokes number. The intrinsic particle density may assume a worst case value of 10 g/cm³:

$$R = U/v$$

$$S_{tk} = \frac{d_p^2 \rho_p U C^*}{18 \eta D}$$

A wall tap sample port can be approximated as a sampling probe oriented 90° to the flow. Calculate the aspiration efficiency:

$$\frac{C}{Co} = \frac{1}{1 + 8 S_{tk} R^{1/2}}$$

Repeat the calculations for particle sizes in the range of interest. When the aspiration efficiency is significantly different from 1, the calculated efficiency should be used to correct the measured particle concentration, C .

R1-4 References

Stevens, D. C. Review of Aspiration Coefficients of Thin-Walled Sampling Nozzles. J. Aerosol Sci., 17(4):729–743, 1986.

Vincent, J. H., D. C. Stevens, D. Mark, M. Marshall, and T. A. Smith. On the Aspiration Characteristics of Large-Diameter, Thin-Walled Aerosol Sampling Probes at Yaw Orientations With Respect to the Wind. J. Aerosol Sci., 17(2):211–224, 1986.

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SEMI F25-0697 (Reapproved 0303)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 OXIDANT SPECIALTY GASES

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published June 1997.

1 Purpose

1.1 The purpose of this document is to set a maximum permissible particle concentration for 10/0.2 grade oxidant specialty gases and to describe a reference method for its verification.

2 Scope

2.1 This document applies only to oxidant gases delivered through specialty gas systems at pressures up to 8×10^5 Pa (8 atmospheres). This method is not suitable for direct sampling from high pressure cylinders at pressures above 8×10^5 Pa (8 atmospheres). This document applies only to the following gas:

Oxygen (O ₂)
Nitrous Oxide (N ₂ O)
Nitrogen Trifluoride (NF ₃)

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

3 Referenced Standards

3.1 SEMI Standard

SEMI C6.2 — Particle Specification for Grade 20/0.02 Oxygen Delivered as Pipeline Gas

3.2 JIS Standard¹

JIS B 9921 — Japanese Industrial Standard (1989), “Light Scattering Automatic Particle Counter”

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

¹ Japanese Industrial Standards, Available through the Japanese Standards Association, 1-24, Akasaka 4-Chome, Minato-ku, Tokyo 107-8440, Japan. Telephone: 81.3.3583.8005; Fax: 81.3.3586.2014 Website: <http://www.jsa.or.jp>

4 Terminology

4.1 Variables

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

4.2 *gas sample volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1×10^5 Pa (1 atmosphere) pressure. Standard cubic feet (SCF) is defined at 21.1°C (70°F) and 1×10^5 Pa (1 atmosphere) pressure.

4.3 *average observed concentration of counts* (\bar{X}_M , \bar{X}_B) — The average concentration of counts, i.e.:

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

4.4 *calculated concentration of particles* (\bar{X}_C) — The concentration of particles in the system gas obtained by correcting the observed concentration in the system gas for the observed concentration in the background, i.e.:

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

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

$$S_M = \sqrt{\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)}}$$

$$S_B = \sqrt{\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)}}$$

NOTE 1: The third is obtained from the first two, i.e.:

$$S_C = \sqrt{S_M^2 + S_B^2}$$

5 Requirements

5.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 6.

5.2 The specification will be considered met if the calculated concentration of particles plus 2 times the standard deviation does not exceed 10 particles per 25 standard liters, i.e.:

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

6 Apparatus

6.1 *Particle Counter* — An instrument suitable for counting particles in compressed oxidant gases at a pressure of up to 8×10^5 Pa (8 atmospheres) with a 50% counting efficiency at 0.2 micrometer or smaller. The counting efficiency should reach 90% at 0.3 micrometer or smaller. The resolving power of the instrument near 0.2 micrometer should be no worse than 10%. The counting efficiency is determined by a calibration at 1×10^5 Pa (1 atmosphere) pressure using polystyrene latex spheres in an inert gas and a reference particle counter with a proven counting efficiency of not less than 95% at 0.2 micrometer.

NOTE 2: Suitable test methods for determining counting efficiency and resolving power are contained in Japanese Industrial Standard JIS B 9921 (1989), "Light Scattering Automatic Particle Counter". More sensitive particle counters result in a higher measured particle concentration.

7 Test Method

NOTE 3: The details of the sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice. Material safety data sheets should be referred to for safe handling of specialty gases.

7.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing nitrogen, believed to be free of particles 0.2 micrometer or more in diameter, through the instrument and recording the

total number of counts. The nitrogen purge assembly for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Count a minimum of 3 equal intervals, each of at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Calculate \bar{X}_B as defined in Section 4. \bar{X}_B must not exceed 2 counts per 25 standard liters.

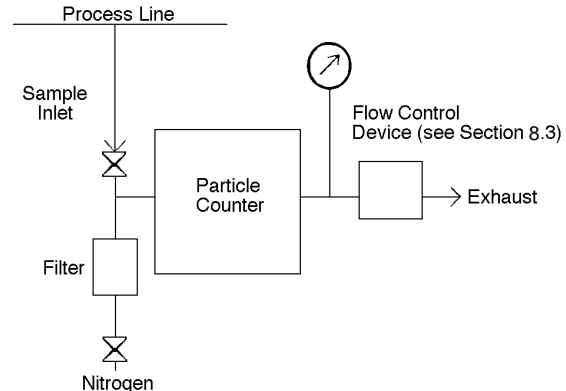


Figure 1
Schematic Diagram of Configuration for
Obtaining Particle Samples from
Oxidant Specialty Gas Systems

7.2 The sampling point should be near the point of use, and sampling lines should be clean, free of combustible substances, and as short as possible.

7.3 The sampling system configuration is shown in Figure 1. The specialty gas system should be connected through a sample valve directly to the particle counter, a pressure gauge, and a flow control device (FCD). When sampling is performed using wall tap sample ports, the aspiration efficiency should be checked using the method described in Related Information 1. The sampling system should contain minimum dead volume and sample tube length. All components in the sampling system should be leak tight and cleaned in accordance with good engineering practice. A leak check of the system should be performed in accordance with normally accepted practice. The FCD may be part of the particle counter. The particle counter should be maintained at the pressure of the gas system. The FCD can be a metering valve and a flow meter or a critical orifice. The exhaust line should be leak tight and piped to an appropriate exhaust system. The exhaust line diameter should be large enough to produce no more than 1×10^4 Pa (1.5 psi) pressure drop during purge and sample flow.

NOTE 4: The alternative sampling configurations described in SEMI C6.2 can also be used for oxygen.

7.4 Using the high purity nitrogen system shown in Figure 1, purge the sampling system and particle

counter for at least 5 minutes at the instrument manufacturer's specified flow rate. The exact purge time should take into account the reactivity of the gas and should be sufficient to purge the entire exhaust line. The purging must be performed before oxidant gas is introduced into the sampling system and after completion of the measurement. The valve leading from the purge nitrogen system should be closed when sampling specialty gases and/or a back flow prevention device should be included in the purge nitrogen system.

NOTE 5: It is known that oxidant gases, although non-flammable, may support combustion. Cleaning in accordance with good engineering practice and nitrogen purging of the sampling system reduce the risk of reaction. The purity of the purge nitrogen should be sufficient to remove reactive substances from the sampling system.

7.5 Count the particles in each of at least 3 equal intervals. Each sample interval must be at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Data obtained during the first 5 minutes after the sample valve is opened may be discarded. Record the number of counts in the sample volume for each interval. Calculate \bar{X}_C and S_C , as defined in Section 4.

NOTE 6: For small volume specialty gas systems, the user and supplier may agree to a smaller sample gas volume than that stated above. The sample point location and the process line pressure and flow rate during the test should be recorded.

8 Report

8.1 The report shall contain the values of all the variables defined in Section 4.

9 Related Documents

Hart, J. J., W. T. McDermott, A. E. Holmer, and J. P. Natwora, Jr. Particle Measurement in Specialty Gases. *Solid State Technol.*, 38(9):111-116, September 1995.

Wang, H. C. and R. Udischas. Counting Particles in High Pressure Electronic Specialty Gases. *Solid State Technol.*, 37(6):97-107, June 1994.

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RELATED INFORMATION 1

NOTES ON ASPIRATION EFFICIENCIES OF WALL TAP SAMPLE PORTS

NOTICE: This related information is not an official part of SEMI F25 and is not intended to modify or supercede the official standard. Determination of the suitability of the material is solely the responsibility of the user.

R1-1 Introduction

R1-1.1 Isokinetic sample probes are usually not provided in speciality gas systems. Particle sampling is usually performed using wall tap sample ports. Wall tap sample ports are oriented 90° to the process line flow as shown in Figure R1-1. Non-isokinetic flow into the sample tube results from the 90° change in flow direction. Additional deviation from isokinesis can result from stream tube contraction or expansion when the velocities U and v are not equal. The effects of non-isokinetic flow on the measurement should be checked. The particle sampling process should be performed with an aspiration efficiency close to 1.

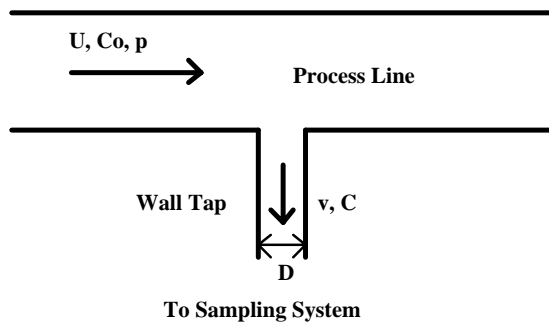


Figure R1-1
Schematic Diagram of Process Line and Wall Tap Sample Port

R1-2 Variables

d_p	Diameter of particle (cm)
ρ_p	Intrinsic density of particle (g/cm ³)
η	Dynamic viscosity of the gas (g/s-cm)
λ_o	Mean free path of the gas at atmospheric pressure (cm)
p	Pressure of the gas (Pa)
P_o	Atmospheric pressure ($= 1 \times 10^5$ Pa)
D	Diameter of wall tap sample tube (cm)
U	Average velocity of the process line flow (cm/s)
v	Average velocity of the sample line flow (cm/s)
C^*	Stokes-Cunningham slip correction factor
S_{tk}	Stokes number
R	Velocity ratio
Co	Particle concentration in the process line (cm ⁻³)
C	Particle concentration in the sample line (cm ⁻³)

R1-3 Calculations

R1-3.1 The Stokes-Cunningham slip correction factor is calculated for particles in the size range of interest:

$$C^* = 1 + 2.492 \frac{p_o \lambda_o}{p d_p} + 0.84 \frac{p_o \lambda_o}{p d_p} e^{-0.435 p d_p / p_o \lambda_o}$$

Calculate the velocity ratio and Stokes number. The intrinsic particle density may assume a worst case value of 10 g/cm³:

$$R = U/v$$

$$S_{tk} = \frac{d_p^2 \rho_p U C^*}{18 \eta D}$$

A wall tap sample port can be approximated as a sampling probe oriented 90° to the flow. Calculate the aspiration efficiency:

$$\frac{C}{Co} = \frac{1}{1 + 8 S_{tk} R^{1/2}}$$

Repeat the calculations for particle sizes in the range of interest. When the aspiration efficiency is significantly different from 1, the calculated efficiency should be used to correct the measured particle concentration, C .

R1-4 References

Stevens, D. C. Review of Aspiration Coefficients of Thin-Walled Sampling Nozzles. J. Aerosol Sci., 17(4):729-743, 1986.

Vincent, J. H., D. C. Stevens, D. Mark, M. Marshall, and T. A. Smith. On the Aspiration Characteristics of Large-Diameter, Thin-Walled Aerosol Sampling Probes at Yaw Orientations With Respect to the Wind. J. Aerosol Sci., 17(2):211-224, 1986.

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SEMI F26-0697 (Reapproved 0303)

PARTICLE SPECIFICATION FOR GRADE 10/0.2 TOXIC SPECIALTY GASES

This specification was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published June 1997.

1 Purpose

1.1 The purpose of this document is to set a maximum permissible particle concentration for 10/0.2 grade toxic specialty gases and to describe a reference method for its verification.

2 Scope

2.1 This document applies only to toxic gases delivered through specialty gas systems at pressures up to 8×10^5 Pa (8 atmospheres). This method is not suitable for direct sampling from high pressure cylinders at pressures above 8×10^5 Pa (8 atmospheres). This document applies only to the following gas:

Ammonia (NH ₃)
Arsine (AsH ₃)
Phosphine (PH ₃)

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

3 Referenced Standards

3.1 Japanese Industrial Standard¹

JIS B 9921 — Japanese Industrial Standard (1989), “Light Scattering Automatic Particle Counter”

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

4 Terminology

4.1 Variables

V_{Mi} =	Volume of the i^{th} sample interval of the system gas
V_{Bi} =	Volume of the i^{th} sample interval of the background
X_{Mi} =	Concentration of particles observed in the i^{th} sample of interval of the system gas

X_{Bi} =	Concentration of particles observed in the i^{th} sample of interval of the background
N_M =	Number of sample intervals of the system gas
N_B =	Number of sample intervals of the background
\bar{X}_M =	Average observed concentration of counts in the system gas sample
\bar{X}_B =	Average observed concentration of background counts
\bar{X}_C =	Calculated concentration of particles in the system gas
S_M =	Standard deviation of \bar{X}_M
S_B =	Standard deviation of \bar{X}_B
S_C =	Standard deviation of \bar{X}_C

4.2 *gas sample volume* (V_{Mi} , V_{Bi}) — The volume of the sample interval, expressed in standard liters at standard conditions, 0°C (32°F) and 1×10^5 Pa (1 atmosphere) pressure. Standard cubic feet (SCF) is defined at 21.1°C (70°F) and 1×10^5 Pa (1 atmosphere) pressure.

4.3 *average observed concentration of counts* (\bar{X}_M , \bar{X}_B) — The average concentration of counts, i.e.:

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

4.4 *calculated concentration of particles* (\bar{X}_C) — The concentration of particles in the system gas obtained by correcting the observed concentration in the system gas for the observed concentration in the background, i.e.:

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

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

¹ Japanese Industrial Standards, Available through the Japanese Standards Association, 1-24, Akasaka 4-Chome, Minato-ku, Tokyo 107-8440, Japan. Telephone: 81.3.3583.8005; Fax: 81.3.3586.2014 Website: <http://www.jsa.or.jp>

$$S_M = \sqrt{\frac{\sum (X_{Mi} - \bar{X}_M)^2}{(N_M - 1)}}$$

$$S_B = \sqrt{\frac{\sum (X_{Bi} - \bar{X}_B)^2}{(N_B - 1)}}$$

NOTE 1: The third is obtained from the first two, i.e.:

$$S_C = \sqrt{S_M^2 + S_B^2}$$

5 Requirements

5.1 *Maximum Permissible Particle Concentration* — 10 particles per 25 standard liters as determined by the instrument specified in Section 6.

5.2 The specification will be considered met if the calculated concentration of particles plus 2 times the standard deviation does not exceed 10 particles per 25 standard liters, i.e.:

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

6 Apparatus

6.1 *Particle Counter* — An instrument suitable for counting particles in compressed toxic gases at a pressure of up to 8×10^5 Pa (8 atmospheres) with a 50% counting efficiency should reach 90% at 0.3 micrometer or smaller. The resolving power of the instrument near 0.2 micrometer should be no worse than 10%. The counting efficiency is determined by a calibration at 1×10^5 Pa (1 atmosphere) pressure using polystyrene latex spheres in an inert gas and a reference particle counter with a proven counting efficiency of not less than 95% at 0.2 micrometer.

NOTE 2: Suitable test methods for determining counting efficiency and resolving power are contained in Japanese Industrial Standard JIS B 9921 (1989), "Light Scattering Automatic Particle Counter". More sensitive particle counters result in a higher measured particle concentration.

7 Test Method

NOTE 3: The details of the sampling configuration, measurement procedure, and instrument calibration procedure and frequency must be agreed upon by the user and supplier, taking into account good engineering practice. Material safety data sheets should be referred to for safe handling of specialty gases.

7.1 Determine the average observed concentration of counts in the background (\bar{X}_B) by passing nitrogen, believed to be free of particles 0.2 micrometer or more in diameter, through the instrument and recording the total number of counts. The nitrogen purge assembly

for performing this test, using a filter which removes particles in this size range, is shown in Figure 1. Count a minimum of 3 equal intervals, each of at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Calculate \bar{X}_B as defined in Section 4. \bar{X}_B must not exceed 2 counts per 25 standard liters.

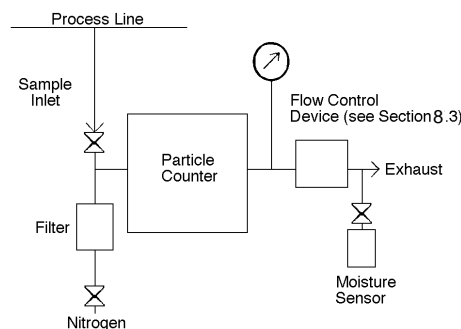


Figure 1
Schematic Diagram of Configuration for
Obtaining Particle Samples from Toxic Specialty
Gas Systems

7.2 The sampling point should be near the point of use, and sampling lines should be clean and as short as possible.

7.3 The sampling system configuration is shown in Figure 1. The specialty gas system should be connected through a sample valve directly to the particle counter, a pressure gauge, and a flow control device (FCD). When sampling is performed using wall tap sample ports, the aspiration efficiency should be checked using the method described in Related Information 1. The sampling system should contain minimum dead volume and sample tube length. All components in the sampling system should be leak tight and cleaned in accordance with good engineering practice. A leak check of the system should be performed in accordance with normally accepted practice. The FCD may be part of the particle counter. The particle counter should be maintained at the pressure of the gas system. The FCD can be a metering valve and a flow meter or a critical orifice. The exhaust line should be leak tight and piped to an appropriate exhaust system. The exhaust line diameter should be large enough to produce no more than 1×10^4 Pa (1.5 psi) pressure drop during purge and sample flow.

7.4 Using the high purity nitrogen system shown in Figure 1, purge the sampling system and particle counter for at least 5 minutes at the instrument manufacturer's specified flow rate. The exact purge time should take into account the reactivity of the gas and should be sufficient to purge the entire exhaust line. A moisture sensor should be placed at the system exhaust to verify less than 1 ppmv water in the purge

nitrogen. The purging must be performed before toxic gas is introduced into the sampling system and after completion of the measurement. The valve leading to the moisture sensor should be closed when toxic gas is being sampled. The valve leading from the purge nitrogen system should be closed when sampling specialty gases and/or a back flow prevention device should be included in the purge nitrogen system.

NOTE 4: It is known that toxic gases may produce injurious or lethal effects. Nitrogen purging of the sampling system reduces the risk of exposure to toxic gases. The purity of the purge nitrogen should be sufficient to remove reactive substances from the sampling system.

7.5 Count the particles in each of at least 3 equal intervals. Each sample interval must be at least 25 standard liters (0.95 SCF), or 30 minutes, whichever is greater. Data obtained during the first 5 minutes after the sample valve is opened may be discarded. Record the number of counts in the sample volume for each interval. Calculate \bar{X}_C and SC, as defined in Section 4.

NOTE 5: For small volume specialty gas systems, the user and supplier may agree to a smaller sample gas volume than that stated above. The sample point location and the process line pressure and flow rate during the test should be recorded.

8 Report

8.1 The report shall contain the values of all the variables defined in Section 4.

9 Related Documents

Hart, J. J., W. T. McDermott, A. E. Holmer, and J. P. Natwora, Jr. Particle Measurement in Specialty Gases. *Solid State Technol.*, 38(9):111-116, September 1995.

Wang, H. C. and R. Udischas. Counting Particles in High Pressure Electronic Specialty Gases. *Solid State Technol.*, 37(6):97-107, June 1994.

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RELATED INFORMATION 1

NOTES ON ASPIRATION EFFICIENCIES OF WALL TAP SAMPLE PORTS

NOTICE: This related information is not an official part of SEMI F26 and is not intended to modify or supercede the official standard. Determination of the suitability of the material is solely the responsibility of the user.

R1-1 Introduction

R1-1.1 Isokinetic sample probes are usually not provided in speciality gas systems. Particle sampling is usually performed using wall tap sample ports. Wall tap sample ports are oriented 90° to the process line flow as shown in Figure R1-1. Non-isokinetic flow into the sample tube results from the 90° change in flow direction. Additional deviation from isokinesis can result from stream tube contraction or expansion when the velocities U and v are not equal. The effects of non-isokinetic flow on the measurement should be checked. The particle sampling process should be performed with an aspiration efficiency close to 1.

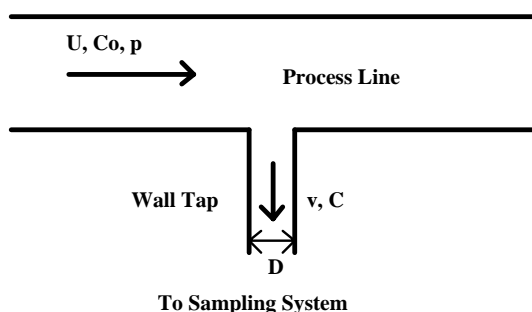


Figure R1-1
Schematic Diagram of Process Line and Wall Tap Sample Port

R1-2 Variables

d_p	Diameter of particle (cm)
ρ_p	Intrinsic density of particle (g/cm^3)
η	Dynamic viscosity of the gas (g/s-cm)
λ_o	Mean free path of the gas at atmospheric pressure (cm)
p	Pressure of the gas (Pa)
P_o	Atmospheric pressure ($= 1 \times 10^5$ Pa)
D	Diameter of wall tap sample tube (cm)
U	Average velocity of the process line flow (cm/s)
v	Average velocity of the sample line flow (cm/s)
C^*	Stokes-Cunningham slip correction factor
S_{tk}	Stokes number
R	Velocity ratio
Co	Particle concentration in the process line (cm^{-3})
C	Particle concentration in the sample line (cm^{-3})

R1-3 Calculations

R1-3.1 The Stokes-Cunningham slip correction factor is calculated for particles in the size range of interest:

$$C^* = 1 + 2.492 \frac{p_o \lambda_o}{p d_p} + 0.84 \frac{p_o \lambda_o}{p d_p} e^{-0.435 p d_p / p_o \lambda_o}$$

Calculate the velocity ratio and Stokes number. The intrinsic particle density may assume a worst case value of 10 g/cm^3 :

$$R = U/v$$

$$S_{tk} = \frac{d_p^2 \rho_p U C^*}{18 \eta D}$$

A wall tap sample port can be approximated as a sampling probe oriented 90° to the flow. Calculate the aspiration efficiency:

$$\frac{C}{Co} = \frac{1}{1 + 8 S_{tk} R^{1/2}}$$

Repeat the calculations for particle sizes in the range of interest. When the aspiration efficiency is significantly different from 1, the calculated efficiency should be used to correct the measured particle concentration, C .

R1-4 References

Stevens, D. C. Review of Aspiration Coefficients of Thin-Walled Sampling Nozzles. J. Aerosol Sci., 17(4):729-743, 1986.

Vincent, J. H., D. C. Stevens, D. Mark, M. Marshall, and T. A. Smith. On the Aspiration Characteristics of Large-Diameter, Thin-Walled Aerosol Sampling Probes at Yaw Orientations With Respect to the Wind. J. Aerosol Sci., 17(2):211-224, 1986.

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SEMI F27-0997 (Reapproved 1103)

TEST METHOD FOR MOISTURE INTERACTION AND CONTENT OF GAS DISTRIBUTION SYSTEMS AND COMPONENTS BY ATMOSPHERIC PRESSURE IONIZATION MASS SPECTROMETRY (APIMS)

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 September 16, 2003. Initially available at www.semi.org October 2003; to be published November 2003. Originally published September 1997.

1 Purpose

1.1 This test will determine the quantity of removable moisture and the degree of interaction with trace concentrations of gas phase moisture, of gas distribution systems and components. APIMS is currently the technique of choice for such tests because it is essentially the only commercially available method capable of ppt moisture analysis and because of its superior response time. This method may provide guidelines for the application of other techniques with similar detection limits and response time to APIMS which are not commercially available at this time.

1.2 The results of this test can be used for qualitative ranking of systems and components and can also be used, by a sufficiently sophisticated user, as input for numerical simulation of distribution system behavior.

2 Scope

2.1 *System and Component Types* — This procedure applies to in-line components to be used to contain electronics grade materials in semiconductor gas distribution systems. The following components are expected to yield meaningful results when tested according to the present method: tubing, connectors (fittings), particle filters, valves (check, relief, shut-off, and metering), regulators, flow-through transducers/sensors, mass flow controllers and meters. Components with dead volumes, such as pressure gauges, can be tested according to this method, but the results will be difficult to interpret. Additional criteria besides those considered here need to be developed for testing components with dead volumes.

2.1.1 As gas phase moisture levels are dominated by desorption from surfaces, the procedure is expected to be most useful for components of large surface area such as particle filters and tubing. Components with very small wetted surface areas may have moisture interactions which are too small to measure using this test. Check valves and relief valves can be tested only if their operating parameters are consistent with the test conditions.

2.1.2 Tubing samples must be rather long (3–4 m) to yield useful results, but they can be bent into a U shape in order to be accommodated into a practical test bench. Radii of curvature should be no less than 6 times the internal diameter of the tube and the minimum number of bends should be used. Most ultra-high purity components are currently supplied with metal gasket connectors, so the test bench employed should be fitted with mating connectors. This type of fitting is usually welded to tubing, which is not recommended as it introduces a potential source of uncontrolled variability to the experiments. Instead, a compression fitting on the tube and a suitable adapter should be used. The compression fitting should not be disconnected after initial connection until the test series is over. In this way, deterioration in integrity of the connection can be avoided. The same type of gasket to compression fitting adapter should be used for all tubing samples.

2.1.3 Purifiers require special test procedures and are not addressed here.

2.1.4 Simple systems consisting of components connected in line can be tested by the present method. Complex systems (i.e., those with more than one potential inlet and/or outlet) will show performance which varies depending on the test configuration (which inlets and outlets are chosen). Testing of such systems is not addressed by the current document.

2.2 *Gases* — The procedure will be carried out in nitrogen. The results will provide a ranking with respect to moisture contribution which may be applied with due caution to components intended for use in other gas streams. Other “inert” gases will have different purging characteristics and may dry a component more quickly or slowly. Reactive gases may react chemically with moisture. Considerations relating to corrosion resistance are outside the scope of the present document, although the test procedure may prove useful in corrosion studies.

2.3 *Operating Situations* — Moisture contribution from a component may be the result of contamination arising in its manufacture, or from subsequent exposure to

ambient air or non-dry gas. Thus, it is necessary to consider two main situations:

1. The “initial dry-down” situation, which is determined by the moisture content of the component as received with the effects of manufacturing process and design, surface quality, pretreatment and packaging convoluted together.
2. The “Response to upset” situation, which is determined by the amount of moisture taken up by the component and subsequently released in any exposure after receipt.

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 allows the determination of moisture interactions which can be used, for example, to rank components in order of decreasing moisture interaction. Because different degrees of moisture interaction are desirable in different situations, selecting the “best” components requires consideration of how they will be used in a given distribution system, either qualitatively or through numerical simulation of distribution system behavior.

4 Referenced Standard

4.1 SEMI Standard

SEMI C15 — Test Method for ppm and ppb Humidity Standards

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

5 Terminology

5.1 Acronyms and Abbreviations

5.1.1 *APIMS* — Atmospheric Pressure Ionization Mass Spectrometry

5.1.2 *EPSS* — Electropolished Stainless Steel

5.1.3 *MFC* — Mass Flow Controller

5.1.4 *ppm* — molar parts per million ($\mu\text{mole/mole}$). The same as ppmv.

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

5.1.6 *ppt* — molar parts per trillion (pmole/mole). The same as pptv.

5.2 Definitions

5.2.1 *induction time* — the elapsed time between when humidified gas is input to the test component and when moisture is detected at the moisture analyzer. For a component and test system which are perfectly transparent to moisture the induction time is equal to the residence time of the gas in the system.

5.2.2 *peak height* — the maximum moisture concentration recorded when a moisture input of predefined length and concentration is introduced to a test component.

6 Required Equipment

6.1 *APIMS* — The APIMS used for moisture detection can be of any type. In order to be considered valid, any series of moisture tests shall reference two single point calibrations, one at the beginning and one at the end of the series. These calibrations shall both be made under the same analytical conditions (flow, pressures, plate voltages, etc.) as the tests and the results shall be within 5% of each other. Provided this condition is met, the interval between calibrations may be left to the discretion of the operator; however, it is suggested that calibration should be carried out whenever the equipment is moved and/or every two weeks of operation.

6.2 *Dry Gas and Moisture Generator* — A source of extremely dry nitrogen (less than 100 ppt moisture) and a moisture generator capable of delivering up to 13 slm nitrogen doped with 50 ppb moisture is required. This generator may be the same as used to calibrate the APIMS. The output of the generator shall be verified according to SEMI C15.

6.2.1 Most ultra-high purity components are currently fitted with metal gasket type connectors. The same type of connector should, therefore, be incorporated into the test bench for connection to the test components.

6.3 *Test Blank* — Any series of tests shall include the results of testing a blank. The blank shall be the shortest convenient length (no more than 1 m) of 1/4" electropolished stainless steel (EPSS) tubing with suitable fittings and/or adapters at either end to enable it to be inserted in place of the test specimen.

6.4 Moisture Pulse Generator — A valving arrangement capable of switching rapidly between dry and humidified nitrogen is also required. An example of such a design is shown in Figure 1. In this design, flow is maintained in both the humidified and dry gas lines at all times. By simultaneously switching valves V_1 and V_3 , or V_2 and V_3 , either humidified or dry gas is directed through the test specimen while the other gas stream is directed to vent. Note that there is a bypass loop so that flow can be maintained to the APIMS when the specimen is removed. In this design, all gas lines, but especially those lines between V_1 and V_3 and between V_2 and V_3 , should be as short as possible and constructed of EPSS tubing of high quality. Maintaining gas lines at a constant temperature between 50 and 80°C wherever possible is also recommended.

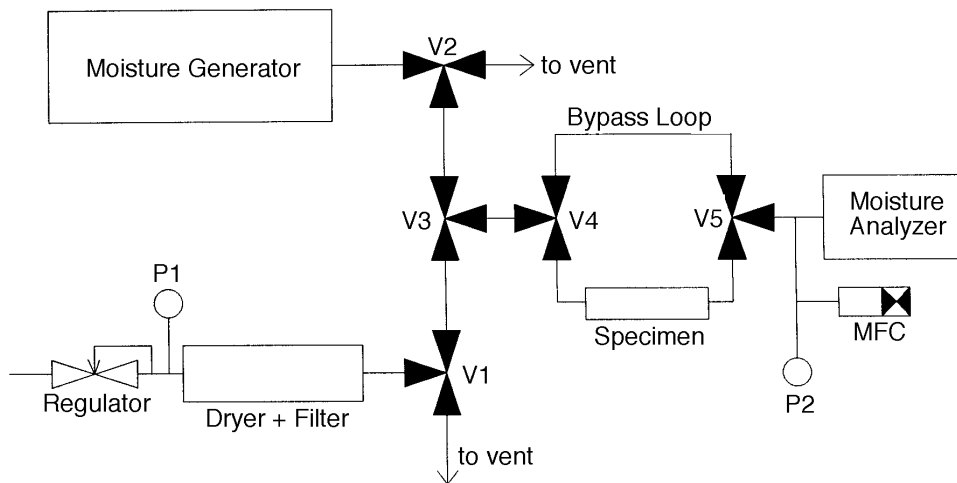


Figure 1
Moisture Test Schematic

6.4.1 The bypass loop will contain a stagnant volume of gas during testing. To avoid exposing the APIMS to a large moisture upset whenever it is fed the gas contained in the bypass loop, the loop should be thoroughly baked out (at $\geq 200^\circ\text{C}$) and protected from atmospheric contamination thereafter. Valve V_4 should be such that some flow can be maintained through the bypass loop and the test specimen simultaneously as well as through each separately. Use of pneumatic valves to facilitate rapid and simultaneous switching is recommended.

6.4.2 Other arrangements than that shown may also be used. The moisture pulse generator should be designed so as to give the fastest possible response of the blank to a change in input moisture level.

6.4.3 Use of a glove box or other such enclosure about the test specimen and adjacent piping, to minimize exposure of the system to ambient contamination when removing and introducing the test specimen, will improve the precision of moisture content measurements but not of moisture interaction measurements. Use of such an enclosure is, therefore, left to the discretion of the operator.

6.5 Temperature Control and Measurement — A stable temperature during the test is of critical importance. The test component shall be kept at 35°C . **TESTS AT DIFFERENT TEMPERATURES CANNOT BE COMPARED.** Ideally, the blank and device under test should be maintained in a temperature-controlled chamber. However, if this is impractical, heating tape can be used. Temperature shall be measured at 1 m intervals on tubing. Measurement at one point is adequate for small components. Temperature control should be to $\pm 1^\circ\text{C}$. A continuous record of temperature during the test shall be maintained.

7 Procedure

7.1 Blank Tests — A blank test shall be carried out after each calibration. If initial dry-down testing of components is not of interest, the initial dry-down test of the test blank may be omitted and the test blank brought to equilibrium with zero gas in whatever manner is most convenient, except that the test blank should not be heated above 200°C . Permanent changes in moisture interaction have been observed at temperatures above this level.

7.1.1 Initial Dry Down — Start the experiment with the blank in place of the test specimen and a flow of dry gas through the APIMS. The APIMS output should be at equilibrium with the lowest moisture level of interest, and in any case no higher than 500 ppt. The flow rate through the test blank should be set according to the Flow Table (see Table 1).

7.1.1.1 Switch the gas flow to pass primarily through the bypass loop while maintaining a small flow through the test blank. Remove the test blank completely from the system. If a glove box or other such enclosure is used, do not remove the test blank from the glove box. Immediately reconnect the test blank to V₄, leaving it disconnected from V₅. Allow dry nitrogen to flow through the test blank for five minutes to purge the air from inside before reconnecting to V₅. Switch the gas to flow only through the blank and not through the bypass loop. The APIMS will show an increase in moisture concentration. Record the APIMS output until it reaches 500 ppt or for 24 hours, whichever is less.

7.1.1.2 Repeat the above test twice for a total of three data sets.

7.1.2 Moisture Input Test — After the APIMS has returned to equilibrium with its initial moisture level, switch the input gas to 50 ppb moisture (by switching valves V₁, V₂, and V₃ simultaneously, if the arrangement of Figure 1 is used) while recording the APIMS response. Maintain this moisture input for 20 minutes. Switch the input to the test blank back to dry nitrogen and record the decrease in moisture level until the initial background is again reached.

7.1.2.1 Repeat the above test twice for a total of three data sets.

7.2 Tests on Actual Specimen

7.2.1 Initial Dry Down — This portion of the test is designed to determine the quantity of removable moisture on the wetted surfaces of a component in the condition in which it is typically supplied. Thus, the results of this test will reflect, by design, any precautions which the supplier has taken to remove moisture and maintain its dryness during shipping.

7.2.1.1 Start the test as in Section 7.1.1. The test specimen should not be unpacked until after the APIMS has equilibrated with the background moisture level. If a glove box or other such enclosure is used, unpack the component in the glove box. Switch the dry nitrogen flow to pass through the bypass loop while maintaining a small component through the test blank. Undo the final layer of packing and any shipping caps or plugs on the component at this point. Remove the sample blank and connect the test specimen to valve V₄ as quickly as possible. Allow the dry nitrogen flow to purge out any

ambient air in the specimen for five minutes, then connect the specimen to V₅. Switch the gas to flow only through the test specimen and not through the bypass loop. The APIMS will show an increase in moisture concentration. Record the APIMS output until it reaches 500 ppt or for 24 hours, whichever is less.

7.2.1.2 After 24 hours, the dry-down part of the test can be terminated. If necessary, the moisture level can be reduced below 500 ppt by baking the component at 200°C or the maximum temperature allowed by the manufacturer for six hours and/or purging at the highest flow of dry gas available. If this is not sufficient to reduce the moisture concentration below 500 ppt, the test may be abandoned.

7.2.2 Moisture Input Test — After the APIMS has returned to equilibrium with its initial moisture level, switch the input gas to 50 ppb moisture (by switching valves V₁, V₂, and V₃ simultaneously, if the arrangement of Figure 1 is used) while recording the APIMS response. Maintain this moisture input for 20 minutes. Switch the input to the test blank back to dry nitrogen and record the decrease in moisture level until the initial background is again reached.

7.2.2.1 Repeat the above test twice for a total of three data sets.

7.2.2.2 In case the moisture level recorded by the APIMS does not reach 50 ppb within 20 minutes, an additional test should be performed in which the moisture input should be continued until equilibrium is reached. Then switch the input to the test specimen back to dry nitrogen and record the decrease in moisture level until the initial background is again reached. This test should be repeated once (two data sets total).

7.2.3 Bake-Out Test — Heat component to maximum bake temperature, according to manufacturer's specifications, for three hours. Allow it to return to room temperature. Continue by repeating the moisture input test, after baking, as in Section 7.2.2.

7.3 Temperature, Flow Rate, and Pressure Specification — The recommended test pressure is 700 kPa (7 bar). However, lower test pressures are appropriate for some models of APIMS. Also, the maximum pressure rating of the test components should not be exceeded. The component temperature shall be maintained at 35 ± 1°C during the test.

7.3.1 In the case of tubing, the test shall be carried out at a single flow determined by the tubing diameter according to the following table. In the case of valves, regulators, mass flow controllers (MFC's), and passive components (gauges, flow meters, and fittings), the test will be carried out at a single flow determined by the table according to the size of the connecting tube stubs.

Regulators should be tested in the fully-open condition (i.e., with the regulator adjusted for minimum pressure drop). The quantity of published data on regulator interaction with moisture is limited, and experiments to investigate the effect of varying the pressure drop across the regulator are encouraged but are not part of this test. MFC's should be tested with a flow control device upstream and the MFC not actively controlling the flow through it (usually referred to as the "purge" setting). It may not be possible to test a given MFC at the specified flow if the flow rating of the MFC is much less than the specified flow. As MFC's are not expected to vary greatly in performance according to their flow rating, MFC's to be tested should generally be chosen to be compatible with the table. If this is not possible, then the MFC should be tested at its rated flow (lower than the flow in the table). This can then be considered a conservative test, as the MFC operated at higher flow would be expected to dry down more quickly.

7.3.2 Particle filters with the same size tubing connectors often have somewhat different sizes and very different flow ratings. In order to be able to make reasonable comparisons between the performance of different filters, it is essential to test filters of similar size at the same flow. However, the flow dependence of the moisture response of particle filters is more complex than that of tubing, and they must be tested at more than one flow in order to model their behavior. In order to meet this requirement and provide data which can be readily compared with the conditions under which the filters can be expected to be used in practice, filters shall be tested at two flows: The first, according to the size of the connecting tube stubs, the second, to be 25% of the rated flow of the filter or 50 slm, whichever is less.

7.3.3 For systems of components, the system shall be tested at the lowest flow of those determined for each component in the system considered separately. If the system includes a filter, it shall be tested at that flow and additionally, at 25% of the rated flow of the filter or 50 slm, whichever is less.

Table 1 Flow Rate

<i>Tubing Outer Diameter (nominal)</i>	<i>Flow (all components)</i>	<i>Flow for Second Test on Filters</i>
$\geq 1/8"$, $< 1/2"$ (≥ 3.2 mm, < 12.7 mm)	6 slm	25% of rated flow or 50 slm whichever is less
$\geq 1/2"$ (≥ 12.7 mm)	13 slm	

8 Reporting Results

8.1 Complete moisture response curves for all test specimens and relevant test blanks should be included.

A summary sheet may compare components in terms of "induction time," peak height, and/or decay time. Temperature, pressure, and flow measurements, and as complete a record as possible of all experimental variables should be noted.

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.

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SEMI F28-1103

TEST METHOD FOR MEASURING PARTICLE GENERATION FROM PROCESS PANELS

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 September 25, 2003. Initially available at www.semi.org October 2003; to be published November 2003. Originally published September 1997.

1 Purpose

1.1 The purpose of this document is to define a method for testing process panels intended for installation in high-purity gas distribution systems. Application of this test method is expected to yield comparable data among process panels tested for the purposes of qualification for this installation.

1.2 This document describes a test method designed to draw comparisons of particulate generation performance of process panels. This test method evaluates the cleanliness of process panels in the “as received” condition as well as under normal operating conditions. The “as received” test is intended to enable the user to evaluate the fabrication, cleaning, and packaging techniques of the manufacturer of the process panel. The test under actual operating conditions is intended to allow the user to evaluate the manufacturer’s component selection as well as the quality of the panel design. The specific flow rates described in both test methods are representative of relatively high flow conditions for a typical process panel.

2 Scope

2.1 This test method addresses total particle counts greater than the minimum detection limit (MDL) of the particle counter and does not consider classifying data into various size ranges.

2.2 This procedure utilizes a particle counter applied to process panels typically used in semiconductor applications. It applies to process gas supply systems (e.g., gas cabinets) which include a process panel, an inert purge panel, and a system vent. Both automatic and manual process panels are within the scope of this test procedure. Panels, as defined in this test method, are considered to consist of 6.35 mm O. D. × 0.89 mm wall (1/4" O.D. × 0.035" wall) tubing and 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 This test method specifies flow and mechanical stress conditions considered typical of conditions which would be expected under moderately aggressive operating conditions. These conditions should not exceed those recommended by the manufacturer. Actual performance under operating conditions at lower flow rates or less aggressive conditions may differ. This test method does not address particle generation under vibrating conditions.

3.2 The test medium is limited to nitrogen, argon, or clean dry air (CDA). Performance with other gases may differ.

3.3 The accuracy of the data generated by this method is limited to the accuracy of the particle measuring instruments utilized.

3.4 This method is written with the assumption that the operator understands the use of the apparatus at a level equivalent to six months of experience.

3.5 This document is not intended as a methodology for monitoring on-going particulate performance once a particular process panel has been tested. Also, this method does not include extended dynamic particle count testing (particle generation after thousands of cycles).

3.6 Auto-crossover systems are not within the scope of this test procedure.

NOTE 1: It should be mentioned that test results from panels equipped with final outlet filters can differ significantly from test results from panels without filters.

4 Referenced Standards

4.1 ISO Standards¹

ISO 14644-1 — Cleanrooms and associated controlled environments Part 1: Classification of air cleanliness.

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

ISO 14644-2 — Cleanrooms and associated controlled environments Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1.

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *CDA* — Clean, dry air

5.1.2 *EP* — Electropolished

5.1.3 *LPC* — Laser particle counter

5.1.4 *psi* — Pounds per square inch

5.1.5 *psia* — Pounds per square inch absolute

5.1.6 *psid* — Pounds per square inch differential

5.1.7 *psig* — Pounds per square inch gauge

5.1.8 *scfm* — Standard cubic feet per minute

5.1.9 *slpm* — Standard liters per minute

5.1.10 *SL* — Standard liters

5.2 Definitions

5.2.1 *background counts* — counts contributed by the test apparatus (including counter electrical noise) with a spool piece in place of the test object.

5.2.2 *Compressed Gas Association (CGA)* — also frequently used to refer to a fitting, as defined and specified by the Compressed Gas Association, which is used to connect a gas source cylinder to a panel inlet.

5.2.3 *condensation nucleus counter (CNC)* — a discrete particle counting instrument that detects particles, in a gaseous stream, by measuring light scattered from droplets grown to measurable size by condensation of supersaturated vapor upon the particles.

5.2.4 *dynamic test* — a test performed to determine particle contribution as a result of valve actuation or regulator adjustment in a process panel during normal operation.

5.2.5 *process panels* — a gas source control piping system for delivering process gases as defined in SEMI F13.

5.2.6 *sample flow rate* — the volumetric flow rate drawn by the particle counter for particle detection.

5.2.7 *sampling time* — the time increment over which counts are recorded.

5.2.8 *source pressure* — pressure of the source gas applied to the cylinder connection at the panel inlet.

5.2.9 *spool piece* — a null component consisting of a straight piece of electropolished tubing and appropriate fittings used in place of the test component to establish the baseline.

5.2.10 *standard conditions* — 101.3 kPa, 0.0°C (14.73 psia, 32°F).

5.2.11 *static test* — a test performed on an as-received process panel with all valves in the fully-open position.

5.2.12 *test duration* — total time required to complete the test procedure.

5.2.13 *test flow rate* — volumetric flow rate of the test gas at standard conditions as defined in Section 5.2.10.

5.2.14 *test pressure* — pressure immediately downstream of the test panel.

6 Test Procedure

6.1 Test Conditions

6.1.1 *Precautions* — This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address the safety considerations associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations before using this method.

6.1.1.1 Exhaust from the CNC may contain hazardous and/or flammable vapors and should be properly treated before being released to atmosphere.

6.1.2 The test is to be conducted at a normal indoor temperature of between 18°C (64°F) and 26°C (78°F).

6.1.3 Test apparatus must be enclosed in a Class 100 environment (per ISO 14644). Use procedures necessary to maintain Class 100 when handling test apparatus and test components.

6.1.4 Care should be taken to protect the test apparatus from excessive vibration. For example, vacuum pumps and compressors should be isolated from the system.

6.2 Apparatus and Materials

6.2.1 *Test Gas* — Clean, dry nitrogen or air is to be used [minimum dryness –60°C dew point at 790 kPa (100 psig) and < 10 ppm total hydrocarbons]

6.2.2 *Filters* — Filters are required to provide “particle-free” test gas. Each filter must be nine-log retentive, per manufacturer’s specifications, to 0.02 µm particles and have a pressure drop of less than 6.9 kPa (1 psid) at the specified test pressure and flow rate. The filter must be capable of achieving less than 1 particle³

($\geq 0.02 \mu\text{m}$) per cubic foot of test gas under test conditions.

6.2.3 Pressure Regulator — Pressure regulators are required to maintain system test pressures.

6.2.4 Pressure Gauge — Pressure transducers or gauges are required to monitor system test pressures.

6.2.5 Flow Control Device — A 0–100 slpm flow control device is required for controlling the test flow rate.

6.2.6 Tubing — A 6.35 mm O.D. \times 0.89 mm wall (1/4" O.D. \times 0.035" wall) tubing is required. The tubing shall have an average surface roughness of 0.25 micrometer (10 microinch) Ra or less. Electropolished tubing (as defined in SEMI F16) is recommended.

6.2.7 Sampler — The sampling system consists of a diffuser equipped with a pressure reducing device (e.g., critical orifice) and a sample probe. All parts of the sampling system are to be electropolished with a surface roughness of 0.25 micrometer (10 microinch) Ra or less. If a critical orifice is to be used for pressure reduction, it is not required to be electropolished. The diffuser and sample probe must be sized to obtain isokinetic sampling at the specified test conditions. The critical orifice must be sized to obtain the specified panel pressure at the specified test flow rate.

6.2.8 Spool Piece — Spool pieces shall be electropolished 316L stainless steel tubing with a surface

finish of 0.25 micrometer (10 microinch) Ra or better. The spool piece is to be installed in the system in place of the test device while obtaining background counts for the system.

6.2.9 Fittings — Use face seal connectors or compression fittings depending on test panel end connections.

6.2.10 Gaskets — Use non-plated metal gaskets where required.

6.2.11 A CNC capable of detecting particles at least as small as $0.02 \mu\text{m}$ at a 50% efficiency envelope as defined by ISO 14644, with a sampling flow rate of 1.23 SLPM (0.05 scfm) or greater, is to be used for particle counting. A laser particle counter, capable of detecting particles at least as small as 0.1 micro meters, may be used in addition to, or in place of, the CNC. Test durations in this test method have been established based on a sampling flow rate of 1.23 SLPM (0.05 scfm).

6.2.11.1 Instruments should be calibrated regularly, according to manufacturer's recommendations. For the CNC, this includes routine checks of sample flow rate, working fluid level, and zero.

6.2.11.2 The CNC and data collection equipment should have power surge suppression.

6.2.11.3 Setup and Schematic — See Figure 1.

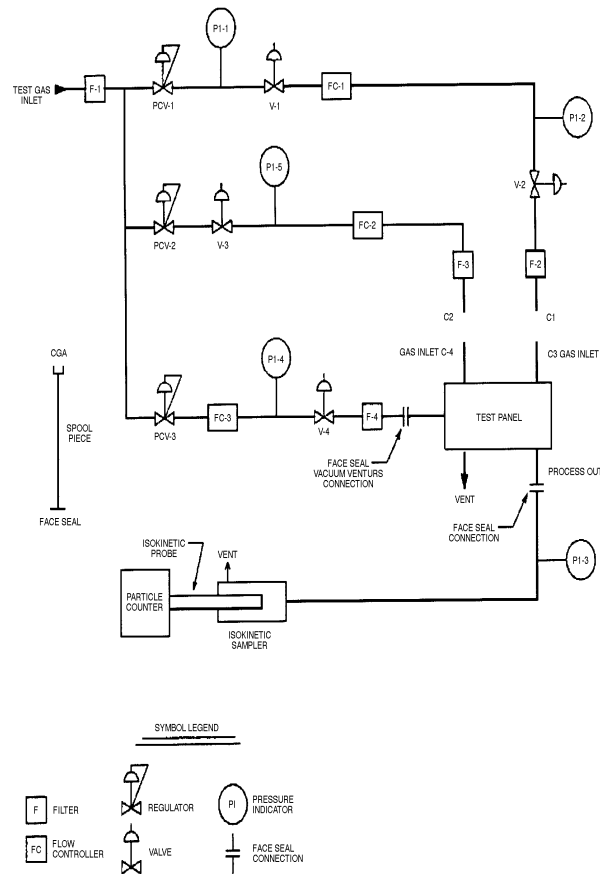


Figure 1
Panel Test Apparatus

6.2.11.4 The spool piece is to be installed when the test stand is not in use. A continuous low flow is to be maintained to purge the system. The particle counter may be turned off. For an extended shutdown, the system (excluding the CNC) should be pressurized and capped.

6.2.11.5 After initial construction, the spool piece should be installed and the system should be cleaned up by running a high-flow rate of test gas (> 60 SLPM) and gently tapping all components (except the CNC) downstream of the final filter. This procedure should be followed by a start-up phase which characterizes system cleanliness by conducting the entire test protocol with the spool piece installed.

6.3 Test Procedures

NOTE 2: Ensure the counter is counting continuously and reporting data each minute. For the duration of the test, the counter should be continuously counting, except where noted in the test protocol.

6.3.1 This test method classifies panels into three pressure ranges: high, medium, and low. The test pressures and flow rates for each pressure classification are given in Table 1 below.

Table 1 Panel Test Conditions

<i>Panel Pressure Class</i>	<i>High</i>	<i>Medium</i>	<i>Low</i>
Inlet Supply Pressure	3450 KPa (500 psig)	550 KPa (80 psig)	210 KPa (30 psig)
Panel Outlet Pressure	210 KPa (30 psig)	210 KPa (30 psig)	0 KPa (0 psig)
Test Flow Rate	60 slpm	60 slpm	5 slpm

6.3.1.1 The test flow rates stated in the above table are meant to serve as a general guideline. The actual test flow rates may vary depending on the maximum panel flow rate as recommended by the manufacturer. The maximum test flow rate should be the manufacturer's maximum recommended flow rate. If no such maximum flow rate is recommended by the manufacturer, the flow rates in the above table should be used.

6.3.2 *Background Test* — Ensure that all valves depicted in Figure 1 are in the closed position. Connect the spool piece to the purge gas inlet (C-2) and the isokinetic sampler inlet. Ensure that the spool piece and proper adapters are in place on the test apparatus. (C-1 should be capped at this time.)

6.3.2.1 Close valve V-3. Adjust the regulator (PCV-2) until PI-5 indicates the appropriate pressure in Table 1. Open valve V-3 and use FC-2 to establish the specified test flow rate listed in Table 1. Measure the static background count. Background count is established when the counter has sampled a minimum of 74 SL (3 scf), and the arithmetic average during the last 74 SL (3 scf) of gas sampled is < 71 particles/m³ (< 2 particles/scf). At a sample flow rate of 1.23 SLPM (0.05 scfm), the time required is one hour. Ensure that the background counts are stable or decreasing. If background cannot be achieved after 147 SL (6 scf) have been sampled, there may be a problem with the counter or test apparatus.

6.3.2.2 Disconnect the spool piece from C-2 and connect it to C-1. Cap C-2 at this point.

6.3.2.3 Open valve V-1 and close valve V-2. Adjust the regulator (PCV-1) until PI-2 indicates the appropriate supply pressure listed in Table 1. Open valves V-1 and V-2 to establish flow. Using the flow control device (FC-1) set the appropriate test flow rate listed in Table 1. Measure the static background count. Background count is established when the counter has sampled a minimum of 74 SL (3 scf), and the arithmetic average during the last 74 SL (3 scf) of gas sampled is < 71 particles/m³ (< 2 particles/scf). At a sample flow rate of 1.23 SLPM (0.05 scfm), the time required is one hour. Ensure that the background counts are stable or decreasing. If background cannot be achieved after 147 SL (6 scf) have been sampled, there may be a problem with the counter or test apparatus.

6.3.2.4 Actuate valve V-2 at 1 cycle per minute to measure the background counts under dynamic test conditions. Dynamic background count is established when the counter has sampled a minimum of 74 SL (3 scf), and the arithmetic average during the last 74 SL (3 scf) of gas sampled is < 106 particles/m³ (< 3 particles/scf). At a sample flow rate of 1.23 SLPM

(0.05 scfm), the time required is 1 hour. If dynamic background cannot be achieved after 147 SL (6 scf) have been sampled, there may be a problem with the counter or test apparatus.

6.3.3 *Static Test* — Using the flow control device (FC-1), decrease the flow rate to 2.5–5.0 SLPM (0.1–0.2 scfm), so that some flow remains in the system while the test component is installed.

6.3.3.1 Remove the spool piece by first disconnecting the downstream fitting and then the upstream fitting. Immediately install the test panel with all of the process valves in the fully-open position by first connecting the process gas inlet (C-3) fitting and then the process outlet fitting. Uncap C-2 and connect it to the purge gas inlet (C-4) connection of the panel with V-3 in the closed condition. In order to minimize atmospheric contamination and prevent the counter from cooling off, removal of the spool piece and installation of the test panel should take no longer than 2 minutes. Extreme care should be taken to minimize contamination of the test apparatus during this operation. The test panel is to be removed from its inner bag in the Class 100 test area. If the process outlet connection of the test panel has mechanical fittings, these fittings are to be properly connected. If the process outlet has butt weld tube stubs, the connection is to be made with clean compression fittings. Do not permanently crimp any ferrules onto the tube stubs. Nylon ferrules are acceptable. The purge gas inlet connection (C-4) should also be connected at this time. The outlet vent connection should be capped at this time. If this connection is not capped by the manufacturer, it should be covered with Aclar or Nylon 66 squares and taped so as to insure against contamination by the immediate environment. If applicable, all pneumatic valves should be connected using the maximum recommended actuator pressure as specified by the manufacturer.

6.3.3.2 Adjust the panel's process regulator to obtain the appropriate process outlet pressure as indicated in Table 1. Using the flow control device (FC-1), increase the flow to obtain the appropriate test flow rate as indicated in Section 6.3.1.1

6.3.3.3 Turn on the counter and conduct the steady state test. All of the valves on the process panel are to be tested in a fully-open position until 74 SL (3 scf) of gas have been sampled. Cumulative data should be recorded at one-minute intervals.

NOTE 3: The flow rates in Table 1 represent the maximum test flow rates. If a more thorough test is desired, the static portion of the test should be repeated at intervals of 20%, 40%, 60%, and 80% of the full test flow rate. Testing at the lower flow rates may in some instances be more indicative of panel performance under actual use conditions.

6.3.4 Dynamic Test — This test is to immediately follow the static test. To conduct the dynamic test, actuate the panel's final process outlet valve at the rate of 1 cycle/minute for 60 minutes. Each cycle consists of having the valve in the open position for 58 seconds and in the closed position for 2 seconds.

6.3.5 Cylinder Change Test — Close V-2, open V-3 and V-4, and adjust PCV-2 until PI-5 reads 3450 Kpag (500 psig). Adjust the flow rate and pressure to the vacuum venturi using FC-3 and PCV-3. The flow rate and pressure should be set to the manufacturer's recommended values. Next perform a simulated cylinder change purge and evacuation sequence following the valve sequencing, dwell times, pressure settings, etc. as specified by the manufacturer.

6.3.6 Final Steady State Test — Close V-4, V-3, and open V-2. Repeat the step in Section 6.3.3.3.

6.3.7 Unregulated Flow Test (optional) — In some instances, it may be desirable to test the panel with its regulator in the fully-open position. Such a test will give an indication of panel particulate performance without the additional contribution of particles from an operating regulator. In this instance, steps 6.3–6.3.5 should be repeated with the regulator in the wide open condition.

6.4 Data Analysis and Reporting — The following test conditions are to be reported in the data presentation:

1. Date and time of test
2. Operator
3. Test flow rate
4. Test pressures
5. Panel type, manufacturer, and serial number
6. Particle counter manufacturer, serial number, sample flow rate, model number, specified particle size sensitivity, and calibration date
7. Test gas type and dew point
8. A schematic of the test apparatus, including manufacturer's and model numbers of all test apparatus components
9. Calibration dates for all instrumentation.

6.4.1 Data Presentation — Graph the static and dynamic portions of the test separately as counts/minute (measured by the counter) versus time, including the appropriate background (measured with the spool piece in place) with each. Also graph the entire data set as counts per minute versus time.

6.4.1.1 Present the entire raw data set in tabular form.

6.4.2 Data Reduction — The statistical analysis is based on the assumption that the particles generated are randomly distributed and are statistically independent of each other. The background counts are independent of the particle performance of the component. The particle counts observed from the test include the counts from the component and the background count. Let:

$$\bar{X}^B = \text{average background particle count}$$

$$\bar{X}^t = \text{average total particle count from test}$$

$$\bar{X}^c = \text{average particle count generated by the test component}$$

Therefore,

$$\bar{X}^t = \bar{X}^c + \bar{X}^B$$

6.4.2.1 A statistical analysis of the data is performed to determine the mean, standard deviation, and the standard error for each particle size range.

6.4.2.2 The sample mean, \bar{X} , or average of the data for each state is given by:

$$\bar{X} = \sum_{i=1}^n \frac{X_i}{n}$$

where X_i is the observed counts for the state and n is the number of samples.

6.4.2.3 The sample standard deviation is a measure of the variability of the data about the mean. The standard deviation, s , for each state is expressed as:

$$s = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n - 1}}$$

6.4.2.4 The standard error, Se or standard deviation of the sampling distribution of the mean for each state is given below:

$$Se = \frac{s}{\sqrt{n}}$$

6.4.2.5 The average particle count for the total number of particles generated, or the sample mean, is an estimation of the population mean. For a 95% confidence level, the population mean, μ_p , will be within two standard errors of the sample mean, or:

$$\mu_p = \bar{X} \pm 2 Se$$

6.4.2.6 In order to determine if there is statistical evidence of the component having an effect on the observed particle count, a test must be conducted to see if the actual difference between the average background count, \bar{X}_B , and the average test count, \bar{X}_t , exceeds two standard errors in a distribution of differences between means. The average particle count generated by the test component, \bar{X}_c , is given by:

$$\bar{X}_c = \bar{X}_t - \bar{X}_B$$

6.4.2.7 The standard error in a distribution of differences between means is expressed as:

$$S_{ec} = \sqrt{S_{et}^2 + S_{eB}^2}$$

6.4.2.8 The 95% confidence interval of the test component itself is then determined by:

$$\bar{X}_c \pm 2 S_{ec}$$

6.4.2.9 If this confidence interval includes 0, then this implies that there is not strong statistical evidence of the component having an effect on the observed particle count. If this interval does not include 0, then this implies there is strong statistical evidence that the component does have an effect on the observed particle count.

6.4.2.10 Present in tabular form the average particle count and the associated upper and lower confidence limits (as calculated in Section 6.4.2) for each test state. In addition, present the background test data separately as a distinct element.

7 Related Documents

Agarwal, J. K. and Sem, G. J., "Continuous Flow, Single Particle Counting Condensation Nucleus Counter", *Journal of Aerosol Science*, v.11.4. July 1950: 343–357

Fissan, H. and Schwientek, "Sampling and Transport of Aerosols", *TSI Journal of Particle Instrumentation*, v.2.2. July–December 1987: 3–10

Hinds, W. C. *Aerosol Technology: "Properties, Behavior, and Measurement of Airborne Particles"*, John Wiley & Sons. 1982: 187–194

VanSlooten, R. A., "Statistical Treatment of Particle Counts in Clean Gases", *Microcontamination*, v.4.2. February 1986: 32–38

ANSI/ASME B46.1² — "Specification for Surface Texture - Surface Roughness, Waviness, and Lay"

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² American Society of Mechanical Engineers, Three Park Avenue, New York, NY 10016-5990, USA. Telephone: 800.843.2763 (U.S./Canada), 95.800.843.2763 (Mexico), 973.882.1167 (outside North America) Website: www.asme.org

SEMI F29-0997 (Reapproved 1103) TEST METHOD FOR PURGE EFFICACY OF GAS SOURCE SYSTEM PANELS

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 September 3, 2003. Initially available at www.semi.org September 2003; to be published November 2003. Originally published September 1997.

1 Purpose

1.1 This document defines the purge efficacy test method recommended for determining the minimum acceptable level of purge efficacy for gas source systems used in semiconductor manufacturing. It is also intended as an aid to the procurement of gas source equipment.

NOTE 1: Users of this specification are encouraged to submit suggested revisions or other comments to SEMI.

2 Scope

2.1 This specification applies to gas source equipment used in semiconductor manufacturing facilities and comparable research and development areas. It includes contamination testing requirements for gas source systems.

2.2 The tests covered by this document are as follows:

2.2.1 Purge efficacy with a non-interactive gas using manufacturers' standard purge sequence.

2.2.2 Purge efficacy with a non-interactive gas using the test method specified purge sequence.

2.2.3 Purge efficacy with an interactive gas using manufacturers' standard purge sequence.

2.2.4 Purge efficacy with an interactive gas using the test method specified purge sequence.

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 The document is not intended to determine the safe operation of a gas source system. The test method described here is for determining the removal efficacy of hazardous gas from the pigtail portion of the gas panel during routine cylinder change operations (pre-purge). It does not address the purge condition after the change of the cylinder (post-purge) nor the purge of the entire gas source system which is usually performed

following its initial installation or during maintenance (maintenance purge).

3.2 Because the response time of moisture and oxygen analyzers utilizing different technologies can differ significantly, this can lead to the possibility that different instruments will report different peak heights when measuring identical contaminant spikes. Therefore, results of tests should only be compared when similar analyzers are used.

4 Referenced Standards

None.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *ppb* — Molar parts per billion, same as ppbv.

5.1.2 *ppm* — Molar parts per million, same as ppmv.

5.2 Definitions

5.2.1 *clean dry air (CDA)* — filtered air filtered to 0.02 mm and dried to a dew point of at least -80° C.

5.2.2 *dry down* — removal of residual moisture in a gas delivery or distribution system, often accomplished by flowing a stream of high purity dry inert gas continuously through the system for an extended period of time.

5.2.3 *dwelt time* — the time for which vacuum or pressure is applied during the evacuation or pressurization steps of a purge operation.

5.2.4 *interactive gas* — a gas that will readily adsorb to the surface of a vessel used to contain or transport it. Examples of interactive gases are hydrogen chloride and moisture.

5.2.5 *non-interactive gas* — a gas that will not adsorb to the surface of a vessel used to contain or transport it. Examples of non-interactive gases are oxygen and nitrogen.

5.2.6 *pigtail* — the pigtail is the part of the gas source equipment that is the flexible connection between the cylinder and the gas panel.

5.2.7 *pigtail bleed* — a pigtail bleed is a reverse flow of purge gas from the pigtail to minimize atmospheric intrusion into the gas panel.

5.2.8 *purge cycle* — following the initial process vent step, a cycle is defined as a pressurization step followed by an evacuation step.

5.2.9 *purified nitrogen* — nitrogen purified to meet the following characteristics:

Moisture	< 20 ppb
Oxygen	< 10 ppb
Total hydrocarbons	< 1 ppm
CO ₂	< 1 ppm
CO	< 1 ppm

6 Ordering Information

6.1 Orders for equipment or services requiring purge efficacy testing in accordance with this test method shall include the following:

6.1.1 This test method number and date of issue.

6.1.2 Test requirements as defined in the detailed specifications for the specific product being purchased.

6.1.3 Whether certifications of tests and a report of the test results is required.

7 Requirements

7.1 *Personnel Qualification* — Personnel performing tests in accordance with this specification shall have suitable training and experience. Such personnel should, as a minimum,

7.1.1 be trained and experienced in operation of gas source control equipment,

7.1.2 be trained and experienced in the use of oxygen and moisture analyzers, and

7.1.3 be familiar with the operation and calibration of the specific equipment used in performing the tests.

8 Apparatus

8.1 *Oxygen Analyzer* — The oxygen analyzer should meet or exceed the following characteristics:

Limit of Detection	10 ppb
Accuracy	± 10 ppb

8.2 *Moisture Analyzer* — The moisture analyzer should meet or exceed the following characteristics:

Limit of Detection	10 ppb
Accuracy	± 10 ppb

8.3 *Pressure Transducer* — The transducer should be of a high-purity type with minimal dead volume and meet or exceed the following characteristics:

Accuracy	± 3.4 kPa (0.5 psi)
Hysteresis	± 3.4 kPa (0.5 psi)
Non-Repeatability	± 0.7 kPa (0.1 psi)

8.4 *Mass Flow Controller or Flow Meter* — The mass flow controller or flow meter should be accurate to ± 0.2 slpm.

8.5 *Moisture Source* — The moisture source must be capable of providing a 2 slpm continuous flow of 2.0 ± 0.2 ppm moisture in nitrogen mixture at 138 kPa (g) [20 psi (g)] for a minimum of 30 minutes.

9 Procedure

9.1 Purge efficacy determination with non-interactive gas at manufacturer's recommended pressure, vacuum, and dwell times.

9.1.1 Connect gas source equipment as in Figure 1.

NOTE 2: To avoid biasing the test results by substantially increasing piping volume to be purged, the dead space between the cylinder connection and the valves isolating the analyzer and the contaminant source must be small compared to the total pigtail volume. The use of mono-block valve and flow-through type pressure transducer is highly recommended.

9.1.1.1 However, the pigtail is connected to the cylinder valve during the purge in an actual cylinder change operation. The small additional volume to be purged in the test setup may be used to account for the volume of the cylinder valve outlet cavity, which must be purged during a cylinder change.

9.1.2 To challenge the gas panel with a known contaminant gas, completely fill and pressurize the gas source system to 207 kPa (g) [30 psi (g)] with CDA as measured at the pigtail with the pressure transducer.

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

9.1.4 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.

9.1.5 Record the pressure and peak oxygen concentration 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.

9.1.6 Repeat Sections 9.1.2–9.1.5 for 2, 5, 25, and 50 purge cycles at the manufacturer’s recommended pressure, vacuum and dwell times. Additional tests using other purge cycle numbers are encouraged and may provide a more complete characterization of the purge performance of the test piece.

9.1.7 Plot the peak residual oxygen concentration versus cycles as in Figure 2 for manufacturer’s recommended pressure, vacuum, and dwell times.

9.2 Purge efficacy determination with non-interactive gas at standard pressure, vacuum and dwell times.

9.2.1 Connect the gas source equipment as in Figure 1.

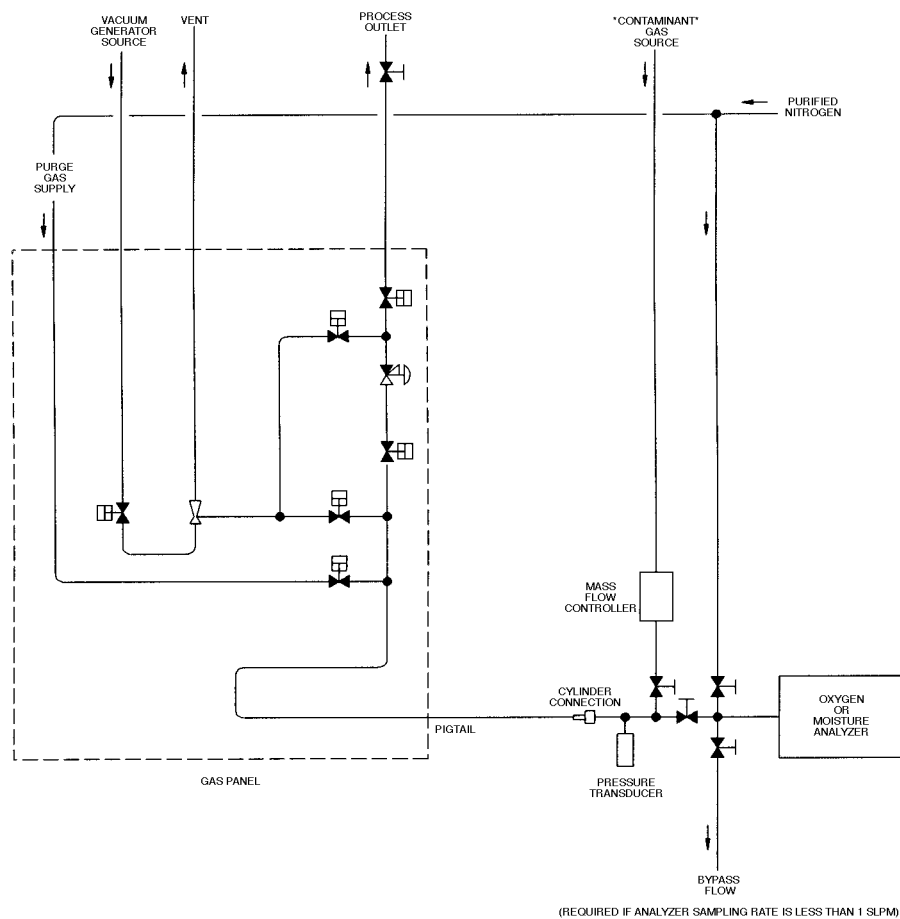


Figure 1
Test Schematic

9.2.2 To challenge the gas panel with a known contaminant gas, completely fill and pressurize the gas source system to 207 kPa (g) [30 psi (g)] with CDA as measured at the pigtail with the pressure transducer.