

## APPENDIX 2

# HEXAVALENT CHROMIUM ALERT

NOTE: This related information is not an official part of this SEMI guide and is not intended to modify or supercede the official guide. It has been derived from the cited document. Publication was authorized by full letter ballot procedures. Determination of the suitability of the material is solely the responsibility of the user.

### A2-1 General

A2-1.1 Hexavalent chromium, which is given off when stainless steel is welded, causes lung cancer in humans. For information about hexavalent chromium, refer to HESIS Hazard Alert, June 1992, Hazard Evaluation System & Information Service, California Occupational Health Program, 2151 Berkeley Way, Annex 11, Third Floor, Berkeley, CA 94704.

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

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

# SEMI F4-1000

## SPECIFICATION FOR PNEUMATICALLY ACTUATED CYLINDER VALVES

This specification 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 August 28, 2000. Initially available on SEMI OnLine September 2000; to be published October 2000. Originally published in 1990; previously published February 2000.

### 1 Purpose

1.1 This specification establishes the minimum design and performance requirements for pneumatically actuated cylinder valves used in semiconductor manufacturing. It is also intended as an aid in the procurement of these valves.

### 2 Scope

2.1 This specification applies to pneumatically actuated valves for use on cylinders containing gases used in semiconductor manufacturing facilities and in comparable research and development areas.

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

### 3 Limitations

3.1 This specification is not a replacement for safety regulations. It is the responsibility of the user to ensure that pneumatically actuated cylinder valves comply with all applicable safety regulations.

3.2 This specification excludes design or performance requirements for any electrically operated devices used in conjunction with, or as a replacement for, pneumatic actuators.

### 4 Referenced Standards

#### 4.1 SEMI Standards

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

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 High Purity Semiconductor Manufacturing Applications

SEMI F32 — Test Method for Determination of Flow Coefficient for High Purity Shutoff Valves

SEMI S5 — Safety Guideline for Flow Limiting Devices

#### 4.2 ASME Document<sup>1</sup>

B1.1 — Unified Inch Screw Threads (UN and UNR Thread Form)

#### 4.3 CGA Standards<sup>2</sup>

CGA G-4.1 — Cleaning Equipment for Oxygen Service

CGA S-1.1 — Pressure Relief Device Standards - Part 1 - Cylinders for Compressed Gases

CGA S-7 — Method for Selecting Pressure Relief Devices for Compressed Gas Mixtures for Cylinders

CGA V-1 — Compressed Gas Cylinder Valve Outlet and Inlet Connections

CGA V-9 — Compressed Gas Association Standard for Compressed Gas Cylinder Valves

#### 4.4 ISO Standards<sup>3</sup>

ISO 68 — General Purpose Screw Threads - Basic Profile

ISO 261 — General Purpose Metric Screw Threads - General Plan

#### 4.5 US Code of Federal Regulations<sup>4</sup>

Title 49 CFR, Parts 100–199 —Transportation

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

### 5 Terminology

#### 5.1 Definitions

5.1.1 *burst pressure* — a pressure at which rupture or uncontrolled leakage of one or more of the pressure retaining components of the device occurs.

1 American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017

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

3 International Organization for Standardization, Case Postale 56, Geneva 20, CH-1211, Switzerland

4 Department of Transportation, Superintendent of Documents, U.S. Government Printing Office, Mail Stop: SSOP, Washington, D.C. 20402-9328

5.1.2 *cylinder* — a pressure vessel designed for pressures higher than 276 kPa (40 psia), having a circular cross-section, and a maximum water capacity of less than 454 kg (1,000 lbs.). It does not include a portable tank, multi-unit tank car tank, cargo tank, or tank car.

5.1.3 *cylinder valve* — a mechanical device attached to a compressed gas cylinder that permits flow into or out of the cylinder when the device is in the open position and prevents flow when in the closed position.

5.1.4 *disconnect shutoff* — a device, which will not permit the flow of gas through an open cylinder valve without attachment to an outlet connector.

5.1.5 *flow coefficient* (Cv) — a numeric constant used to characterize the flow capacity of a valve.

5.1.6 *manual locking device* — a device used to prevent the cylinder valve from opening during transportation or service.

5.1.7 *manual override* — a device used for opening the cylinder valve manually.

5.1.8 *normal temperature* — a temperature of 21°C ± 6°C (70°F ± 10°F).

5.1.9 *normally closed* — a design in which the cylinder valve closure member automatically assumes the closed position upon loss of compressed gas to the actuator.

5.1.10 *packless valve* — a valve with a diaphragm or bellows stem seal instead of a packing or O-ring seal at the stem.

5.1.11 *pneumatic actuator* — a device which converts compressed gas pressure into mechanical motion and force to move the cylinder valve closure member.

5.1.12 *proof pressure* — a pressure greater than the cylinder valve's rated pressure that the cylinder valve can withstand without impairing its ability to meet the leak rate limits specified in SEMI F1 upon return to rated pressure.

5.1.13 *rated pressure* — the pressure at which the cylinder valve can meet the performance and qualification requirements of this specification. Rated pressure shall be specified by the manufacturer.

5.1.14 *valve closure member* — that part of the cylinder valve which is positioned in the flow stream to permit or obstruct flow, depending on its closure position.

5.1.15 *wetted surface* — surface within the interior of the cylinder valve, which is in contact with the controlled gas. The surface in contact with the gas used to actuate the cylinder valve is not, for the purpose of this specification, considered a "wetted surface".

## 6 Design Requirements

6.1 *General* — Components not covered by this specification or CGA Standards shall be constructed such that they do not compromise the safety, substantiality, and durability of the valve or actuator.

6.2 *Configuration* — The cylinder valve shall be a packless valve design with a normally closed actuator.

6.3 *Size* — The cylinder valve and actuator shall be of a design size which permits the use of a protective transportation cover which satisfies the requirements of 49 CFR Section 173.40(d)(2).

6.4 *Materials* — Ferrous materials used for the wetted surfaces shall be per SEMI F20. Other materials may be supplied to satisfy specific material or performance requirements, provided they are compatible with the gas service.

6.5 *Inlet* — The inlet of the cylinder valve shall have tapered external threads as specified by CGA V-1 or other recognized governmental or industry standards for the connection of cylinder valves to gas cylinders.

6.6 *Outlet* — The outlet of the cylinder valve shall conform to a CGA 630/710 series connection in accordance with CGA V-1 or other recognized governmental or industry standards for the outlet connections of gas cylinder valves.

6.7 *Actuator Pressure* — The actuating pressure required to open the cylinder valve fully shall not exceed 550 kPa (gauge) (80 psig).

6.8 *Manual Override* — A manual override shall be supplied.

6.9 *Vents* — Vent hole(s) for mass spectrometer helium leak testing shall be supplied at appropriate locations.

6.10 *Internal Volume* — The wetted surfaces should be configured, and the internal volume minimized, to enhance the efficacy of purging.

6.11 *Particle Control* — The design should minimize accumulation and generation of particles.

6.12 *Threaded Fasteners* — Threaded fasteners used in the covers, housings, casings, and any external mounting brackets shall have threads conforming to, as applicable, the ASME B1.1, ISO 68, or ISO 261 standards.

6.13 *Cleanliness* — The cylinder valve and actuator shall be cleaned, as a minimum, per CGA Standard G-4.1.

## 7 Optional Design Requirements

7.1 *Flow Limiting Device* — Provision for limiting flow, per SEMI S5, shall be supplied when specified.

7.2 *Pressure Relief Devices* — Pressure relief devices must be required in accordance with CGA S-1.1 or S-7.

7.3 *Disconnect Shutoff* — A disconnect shutoff shall be supplied when specified.

7.4 *Manual Locking Device* — A manual locking device shall be supplied when specified.

7.5 *Electropolish* — Cylinder valves shall be electropolished per SEMI F19 when specified.

7.6 *Cleanliness* — Cleaning above the minimum requirements of CGA G-4.1 may be specified.

7.7 *Position Indicator* — A device for visual confirmation of open or closed position may be specified.

## 8 Qualification Requirements

8.1 *General* — Any new cylinder valve design or substantially revised cylinder valve design shall be tested prior to production to demonstrate compliance with this specification.

8.1.1 *Leakage* — Cylinder valves shall be leak tested using the methods and leak rate limits specified in SEMI F1 for qualification of components.

8.1.2 *Operating Temperature Range* — The cylinder valves shall perform satisfactorily at any temperature between -40°C (-40°F) and 55°C (131°F).

8.2 *Qualification Cylinder Valve Samples* — Nine cylinder valves shall be selected at random from the pre-production manufacturing phase of the product for qualification to this specification.

- a. One cylinder valve shall be used for the Flow Coefficient and Transportation Vibration Tests.
- b. Three cylinder valves shall be used for the Pressure Tests.
- c. Three cylinder valves shall be used for the Endurance Test.
- d. Two cylinder valves shall be used for the Tip-Over Tests.

8.3 *Flow Coefficient Test* — The flow coefficient of a fully open cylinder valve tested without a flow restrictor shall be a minimum of 0.1. The method of test for determining the flow coefficient shall be as prescribed by SEMI F32.

8.4 *Transportation Vibration Test* — After being subjected to vibration for one hour at a frequency of

3400 to 3600 vibrations per minute at an amplitude of 0.8 mm (0.031 in.) to 1.6 mm (0.063 in.), the cylinder valve shall not exceed the inboard, outboard, and internal leak rate limits specified.

### 8.5 Pressure Tests

8.5.1 *Back Pressure* — The cylinder valve shall not exceed the internal leak rate limits specified in SEMI F1 with the cylinder valve closed, the rated pressure applied at the outlet, and the inlet connected to the leak detector.

8.5.2 *Proof Pressure* — The proof pressure at normal temperature shall be a minimum of 1.67 times the rated pressure. Tests shall be conducted following the back pressure test with the cylinder valve open, the outlet capped, and the pressure relief port capped or plugged. After exposure to an aerostatic proof pressure for ten minutes, the cylinder valve shall not exceed the inboard, internal, and outboard leak rate limits specified in SEMI F1.

8.5.3 *Burst Pressure* — The burst pressure at normal temperature shall be a minimum of three times the rated pressure. Tests shall be conducted hydrostatically following the proof pressure test with the cylinder valve open, the outlet capped, and the pressure relief port capped or plugged. Visible outboard leakage is not permitted during the burst test. After testing, the cylinder valve need not be functional.

8.5.3.1 *Pressure Rating Determination* — The rated pressure shall be less than or equal to the pressure determined from the burst pressure test as follows:

$$P \leq 0.33BT_m/T_a$$

Where:

P = Rated pressure

B = The lowest burst pressure recorded for the three specimens tested

T<sub>a</sub> = The actual tensile strength of the failed test specimen material obtained from Certified Material Test Reports.

T<sub>m</sub> = The specified minimum tensile strength of the failed specimen material.

NOTE 2: The factor of T<sub>m</sub>/T<sub>a</sub> is included to normalize between the particular steel stock from which the test specimens were made and the minimum tensile strength of the specified steel.

8.6 *Endurance Test* — The cycle life of the cylinder valve shall be at least 5,000 operational cycles. During and after 5,000 operations, the cylinder valve shall not exceed the inboard, internal, and outboard leak rate limits specified in SEMI F1. A cycle shall consist of one opening and one closing of the cylinder valve.

8.6.1 Cylinder valves shall be operated alternately through minimum and maximum temperatures specified in Section 8.1.2 in 1000 cycle increments until completion of the specified number of cycles. The first thermal cycle shall take the cylinder valves to maximum temperature. Cylinder valves shall be at thermal equilibrium while mechanically cycling. Initially, and at the completion of every 1000 cycles, the cylinder valves shall not exceed the inboard, internal, and outboard leakage tests specified in SEMI F1 at normal temperature.

8.6.2 The cylinder valve inlet pressure shall be the cylinder valve rated pressure. Cylinder valves shall be cycled at a rate of one cycle per ten seconds, equally divided between opening and closing. During the off cycle, the cylinder valve's downstream pressure shall be allowed to decay to 50% or less of the inlet pressure.

8.7 *Tip-Over Test* — After being subjected to tip-over tests the cylinder valves shall show no evidence of structural damage, shall not exceed the inboard, internal, and outboard leak rates specified in SEMI F1, and shipping caps or other protective devices shall be able to be removed or adjusted so that the valve can be operated.

8.7.1 Tip-over testing shall be conducted in accordance with CGA V-9, Section 5.2.6.4.

8.7.2 The tip-over tests shall be performed with the cylinder valve installed on a 166 kPa (2400 psig), 250 mm (10 inches) diameter by 1550 mm (61 inches) long (including cap) gas cylinder, or onto a smaller cylinder if the valve is restricted by the manufacture for use on the smaller cylinder.

8.7.3 A shipping cap or other protective device shall be installed onto the cylinder during the tip-over tests. If a special cap or protective device is used it shall conform to the requirements of CGA V-9, Appendix E-49 CFR cylinder drop protocol.

## 9 Manufacturing Requirements

### 9.1 *Acceptance Testing*

9.1.1 Each cylinder valve shall be acceptance tested for inboard, internal, and outboard leakage per SEMI F1.

9.1.2 Each actuator shall be tested for leakage of less than 1 sccm at 550 kPa (80 psig).

9.1.3 Each cylinder valve shall be visually inspected for cleanliness, damage, proper markings, and actuator function, as a minimum.

9.2 *Marking* — Each cylinder valve shall be permanently identified with the following minimum information:

9.2.1 Manufacturer's name or logo.

9.2.2 Manufacturer's part number or part number code.

9.2.3 Outlet type (CGA number or other specification identification).

9.2.4 Pressure relief device marked in accordance with CGA pamphlet S-1.1 or other specification identification.

9.2.5 Manufacturer's serial number or code that will provide traceability for materials, test data, and assembly date.

9.2.6 Flow limiting information when the flow limiting device is integral to the cylinder valve and not intended for removal.

9.3 *Traceability* — The manufacturer shall use a program to qualify raw materials, parts, assemblies, and purchased components and to provide traceability of all components (manufactured or purchased) used in production of cylinder valves.

### 9.4 *Instructions*

9.4.1 Clear, concise instructions and diagrams stated in terms clearly understandable and adequate for proper installation and safe operation shall be provided with each cylinder valve.

9.4.2 Instructions for periodic maintenance or repair, as required, shall be made available by the manufacturer. No repairs or maintenance shall be conducted without these instructions. Parts that require replacement shall be identified.

9.5 *Maintenance* — Ideally, the cylinder valve shall be capable of fulfilling the performance and qualification requirements of this specification after any of the maintenance procedures specified by the cylinder valve manufacturer. Maintenance shall be performed by only those persons the cylinder valve manufacturer deems qualified.

## 10 Related Documents

### 10.1 *Military Specification*<sup>5</sup>

MIL-DTL-2E — Valves, Cylinder, Gas

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<sup>5</sup> Military Specification, Commanding Officer, Naval Publications and Forms Center, Attention: MPFC 105, 5801 Tabor Avenue, Philadelphia, PA 19120

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# SEMI F5-1101

## GUIDE FOR GASEOUS EFFLUENT HANDLING

This guide was technically approved by the Global Environmental Health & Safety Committee and is the direct responsibility of the North American Environmental Health & Safety Committee. Current edition approved by the North American Regional Standards Committee on August 27, 2001. Initially available at [www.semi.org](http://www.semi.org) September 2001; to be published November 2001. Originally published in 1990.

**NOTICE:** This document, as balloted, is intended to replace SEMI F5-90 in its entirety.

**NOTICE:** Paragraphs entitled “NOTE” are not an official part of this guide and are not intended to modify or supersede the official guide. These have been supplied by the Task Force to enhance usage of the guide.

### 1 Purpose

1.1 The purpose of this guide is to provide the semiconductor industry with the general knowledge and background information for understanding the principles of exhaust systems.

1.2 It includes suggestions for layout and selection as well as application of appropriate methods and equipment for abating emissions.

1.3 This guide is intended for use in abatement selection for both gaseous and particulate contaminant materials potentially emitted from semiconductor manufacturing facilities.

### 2 Scope

2.1 *Applicability* — This guide presents a review and evaluation of available information including:

- Chemical and physical properties of chemical compounds and elements, that are, or may be, released from semiconductor manufacturing operations,
- Some current industry practices for separating exhaust systems for different groups of materials,
- Potential methods for abating emissions of gaseous and particulate contaminants exhausted from semiconductor manufacturing operations at “End-of-Pipe”,
- Potential methods for abating emissions of gaseous and particulate contaminants exhausted from semiconductor manufacturing processes at “Point-of-Use” and reasons why they should be used for some processes,
- Standard industry methods of recovery, replacement, and “usage reduction” for minimizing release of materials.

2.2 *Contents* — This document contains the following sections:

1. Purpose
2. Scope
3. Limitations
4. Referenced Standards
5. Terminology
6. Philosophy
7. Classification of Emissions
8. End-of-Pipe Abatement Technologies
9. Point-of-Use (POU) Abatement Technologies
10. Provisions for Emergency Release
11. Summary Table
12. Alternative Approaches
13. Related Documents

Appendix 1 — End-of-Pipe Abatement Design

Appendix 2 — POU Abatement Technology Types

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

### 3 Limitations

3.1 This guide makes only a few recommendations regarding the fire protection aspects of exhaust systems. Additional recommendations on the risk assessment of systems, compatibility of materials, and the potential need for use of non-combustible materials or the fitting of fire detectors, sprinklers etc., are within the scope of other documents.

3.2 Methods for treating solid or liquid waste (created by emission abatement processes) and methods for recovery of gases are not discussed in this document.

## 4 Referenced Standards

### 4.1 SEMI Standards

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

SEMI S5 — Safety Guideline for Flow Limiting Devices

SEMI S8 — Safety Guidelines for Ergonomics Engineering of Semiconductor Manufacturing Equipment

### 4.2 Code of Federal Regulations — United States Environmental Protection Agency<sup>1</sup>

40 CFR Part 63 (Clean Air Act 112 (b)(1))

### 4.3 Uniform Codes<sup>2</sup>

Uniform Fire Code — Article 80 — Hazardous Material

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

## 5 Terminology

### 5.1 Abbreviations and Acronyms

5.1.1 CVD — Chemical Vapor Deposition

5.1.2 EPI — epitaxial deposition

5.1.3 HAP — Hazardous Air Pollutants [as defined by 40 CFR Part 63 (Clean Air Act 112 (b)(1))]

5.1.4 PFC — perfluorocompounds

5.1.5 POU — Point of use

5.1.6 VOC — Volatile Organic Compounds

### 5.2 Definitions

5.2.1 *classification of emissions* — understanding the composition of process exhausts.

5.2.2 *efficiency* — the ratio (or fraction or proportion) of removed chemical species to its input amount

5.2.3 *End-of-Pipe Abatement* — abatement technologies that can be fitted at the discharge point of the exhaust system.

5.2.4 *flow capacity* — the maximum flow any specific equipment can handle.

5.2.5 *occupational exposure limits (OELs)* — for the purpose of this document, OELs are generally established on the basis of an eight-hour workday.

Various terms are used to refer to OELs, such as permissible exposure levels, Threshold Limit Values®, maximum acceptable concentrations, maximum exposure limits, and occupational exposure standards. However, the criteria used in determining OELs can differ among the various countries that have established values. Refer to the national bodies responsible for the establishment of OELs. (Threshold Limit Value is a registered trademark of the American Conference of Governmental Industrial Hygienists.)

5.2.6 *Point-of-Use Abatement* — abatement technologies that can be fitted at the point of discharge of the gaseous emission from semiconductor process equipment. These devices are also called exhaust conditioners.

5.2.7 *removal capacity* — amount of a species which can be removed.

5.2.8 *usage reduction* — reducing the total volume of process chemicals requiring abatement.

## 6 Philosophy

6.1 Gaseous mixtures potentially emitted from numerous processes could also produce hazards within exhaust ducts.

6.2 With increasing legal requirements worldwide on both worker safety and general environmental protection, more effective exhaust management is required.

6.3 This demands a detailed understanding of the chemical and physical properties of the chemical compounds being emitted from processes, and of the conditions under which they are released.

6.4 Careful design of the abatement systems is required to attain both high efficiency and maximum economy.

## 7 Classification of Emissions

7.1 The following fundamental points regarding exhaust emissions should be considered:

- Exhaust contaminants might be solely the material used in the process (e.g., isopropyl alcohol (IPA) from a rinse chamber)
- In plasma and thermal chambers many reactions occur, both those desired and others; therefore by-products are also key components of exhaust emissions.
- In theory, most possible combinations of the elements in the input gases may be found as compounds in the exhaust. In practice, the main components will be the unused process gas (the percentage consumed in the process varies, and

<sup>1</sup> United States Government Code of Federal Regulations, free from the web site at <http://www.epa.gov>

<sup>2</sup> International Conference of Building Officials (ICBO), 5360 Workman Mill Road, Whittier, CA 90601-2298



may be as low as 10%). Additional by-products will have different and sometimes more hazardous properties.

7.2 Considerable research has been conducted into various hazardous by-products of processes, from explosive solids to organochlorine compounds with mutagenic properties. (See Sections 13.1 through 13.3.)

7.3 In considering selection of exhaust systems and abatement technology, it is essential to review all the materials likely to be present in the exhaust.

7.4 Contributions from the materials on the wafer must also be considered, as they often cause blocked exhausts (e.g., aluminum etches produce aluminum chloride (a by-product of removal of aluminum from the wafer with chlorinated gases)).

7.5 Processes that produce specific hazardous by-products should be evaluated for appropriate point-of-use abatement technology before discharge to an exhaust system.

7.6 Gaseous and particulate contaminants emitted from semiconductor manufacturing can be divided conveniently into nine major groups, each of which *might* justify its own exhaust system.

7.6.1 *Group-1 Acid* — Easily hydrolyzable acids, (e.g., hydrogen chloride hydrolyzes into hydrochloric acid.)

7.6.2 *Group-2 Acid aerosols (“difficult-to-scrub”)* — This group includes acids that generate aerosols and that are not easily removed by simple water scrubbers (e.g., aqua regia, sulfuric and nitric).

NOTE 2: High concentrations of easily soluble acids will also fall into the “difficult-to-scrub” category.

7.6.3 *Group-3 Ammonia* — Exhaust streams containing ammonia

7.6.4 *Group-4 Volatile Organic Compounds (VOCs)* — Exhausts containing volatile organic compound vapors or flammable gas discharge requiring destruction.

7.6.5 *Group-5 Pyrophoric* — Exhausts containing pyrophoric gases.

7.6.6 *Group-6 Emergency Release Discharges* — Enclosures are provided with additional exhausts to contain sudden unanticipated releases of gas from cylinders, etc.

7.6.7 *Group-7 Special/Direct* — Additional exhausts used to keep specific gases separate for recovery purposes (e.g., hydrogen (H<sub>2</sub>) reclamation from EPI or PFC reclamation from CVD tools, Oxides of Nitrogen (NO<sub>x</sub>)).

7.6.8 *Group-8 General Exhaust Systems without End-of-Pipe Treatment* — Exhaust of heat or post-treatment exhaust from POU abatement systems.

7.6.9 *Group-9 Highly Toxic Gas Exhaust* — Highly toxic gases especially those with poor warning properties (e.g., arsine, phosphine, diborane, germane). The end user has a primary responsibility to prevent discharge of these gases above a regulated level (e.g., usually one-half (1/2) of Immediately Dangerous to Life and Health (IDLH) levels).

NOTE 3: Facility exhaust systems may be called by a number of different names such as: Acid, Corrosive, Ammonia, Scrubbed, VOC, Solvent, Incinerated, Highly Toxic, Toxic, Dedicated, Special, PFC, General, Heat, Treated, or others. The actual facility exhaust system name, and what groups are routed to each system, will vary by factory and by country.

7.7 The groups listed in Section 7.6 are not a definitive division, but represent a summation of common industry practice for separation. The division is based on three basic principles:

- avoiding mixing of incompatible gases in the exhaust,
- potential need for different end-of-pipe abatement technologies, and,
- keeping materials separate for recovery.

7.8 The number of separate exhaust systems used is entirely a decision for each facility, based on the following:

- processes and chemicals used,
- the number of each type of process,
- whether recovery of gases is required,
- whether point-of-use (POU) abatement is installed, and
- compatibility of emissions with each other or the effluent handling system.

7.9 In theory, if POU abatement systems are fitted to all sources of gaseous effluent all exhaust could then be combined into a single exhaust system.

7.9.1 In practice most facilities operate a compromise between the two extremes with two to four separate exhaust systems and point-of-use abatement systems fitted only to certain sources. The decision (as to which combination to use) will often be based on economic and space constraints.

7.9.2 Safety and environmental factors listed below, should always be considered first when determining specific gaseous sources that require POU abatement systems.

7.10 Many types of process equipment use and exhaust incompatible gases sequentially (e.g., silane and then nitrogen trifluoride (NF<sub>3</sub>) as elements of a deposition and cleaning cycle).

7.10.1 With more than one of these processes being discharged asynchronously into a common exhaust duct, undesired reactions may occur.

7.10.2 It is possible to avoid this problem by fitting the equipment outlet with three-way valves to divert the discharge to the correct exhaust system, depending on equipment process step. Three-way valves may not be necessary if pump ballast-gas flow is increased to dilute gases.

7.10.2.1 Switching must accurately account for the gas transmission times from process chamber inlet to the exhaust valve.

7.10.2.2 Introduction of additional mechanical devices could create major hazards, reliability, and service or maintenance issues.

7.10.2.3 Valves must incorporate failsafe designs to ensure pump outlet is not shut off.

7.10.3 An alternative solution is to fit a local POU abatement system that treats at least one component and is not adversely affected by the other components.

#### 7.11 *Point-of-Use (POU) Abatement Devices*

7.11.1 POU abatement devices are designed to operate at the process tool or pump outlet, and to remove hazardous gases BEFORE they enter the exhaust ductwork.

7.11.2 An end-of-pipe scrubber does not protect the facility from reactions in the ductwork. Reactions between process gases, as discussed above, or reactions with other process by-products within the ductwork itself are not prevented by end-of-pipe systems either.

7.11.3 POU abatement devices may be appropriate wherever the exhaust gases can react in the ductwork.

7.11.4 If process systems are not treated with POU abatement devices, the higher concentrations of hazardous gases will present a higher risk during a leak into the facility space.

7.11.5 Improperly functioning POU abatement devices can create backpressure that can increase the risk of a leak.

7.12 Four significant consequences can be foreseen from effluent gas reactions in the ductwork:

7.12.1 *Blocked Ducts* — Reactions between gases, or condensation of vapors, can produce solids and semi-solids (such as crystals, slurries or gels) that will block the ductwork.

7.12.1.1 Collection of solids can create dead leg sections that may contain reactive byproducts or unused raw materials. Catastrophic release of these trapped materials could generate a significant hazard.

7.12.1.2 Extensive downtime (for clean out, or ducts collapsing under the weight of accumulated solids) is a possible undesirable outcome of this condition.

7.12.1.3 Solids deposited (such as from metal etching) can, themselves, be both corrosive and hazardous, leading to health risks for people performing cleaning in ductwork. (See Related Documents 13.1 through 13.3).

7.12.1.4 Blocked ducts may result in toxic process gases or by products escaping into the work area.

7.12.2 *Duct Corrosion* — Etching and some chamber cleaning process emissions are corrosive. Both metallic (including stainless steel) and plastic ducting can be attacked, resulting in the release of hazardous gas or condensed liquids into the workplace.

7.12.3 *Duct Fires or Explosions* — Flammable and pyrophoric gases can ignite in the ductwork. If the ductwork is combustible or has flammable or combustible deposits in it (e.g., hydrocarbon pump oil), facility fires can result.

7.12.4 Additional information on protection of Industrial Exhaust systems from fires may be found in FM Global Loss Prevention Data Sheet 7-78, "Industrial Exhaust Systems".

NOTE 4: Destruction of whole production lines with multi-million dollar cost impact has resulted from such incidents.

7.12.5 *Formation of Ammonium Compounds* — If ammonia is discharged into a duct with acid compounds, a sub-micron ammonium compound fume can be generated, that is not easily removed by end-of-pipe systems.

7.12.5.1 The result is visible discharges.

7.12.5.2 Ammonium compounds produce solids in the duct.

7.13 If any of the above reactions can occur in the process exhaust, POU abatement should be considered.

7.14 Exhausts of oil-lubricated vacuum pumps should be fitted with well-maintained oil mist separators/filters.

7.15 POU abatement devices may also be needed upstream of the recovery device for the recovery of some gaseous compounds (e.g., hydrogen (H<sub>2</sub>), PFC gases).

## 8 End-of-Pipe Abatement Technologies

### 8.1 Group 1 — Acid Abatement

8.1.1 The appropriate abatement technology for this category is commonly called "wet scrubbing."

8.1.2 Many, if not all, of the semiconductor processes have traditionally been exhausted through centralized wet scrubbers.

8.1.2.1 This procedure has limited technical acceptability and may not meet the requirements of all regulatory agencies.

8.1.2.2 This technology should be targeted at abatement of acid gases, vapors, mists, and fumes emitted from CVD, dry etching, and wet chemical operations.

8.1.2.3 Most wet chemical processes are typically carried out under local exhaust ventilation devices, such as fume hoods, or in specially designed rooms that are totally exhausted. These types of operations include:

- Processes used to clean wafers or clean wafer handling equipment (such as quartz components, tubing),
- Processes used in wafer fabrication for surface treatment such as oxide, silicon nitride and other surface treatments, (e.g., metal etching), and
- Processes used for development of positive photoresist.

8.1.2.4 The chemical species emitted depends on both the chemicals used in the processes and the conditions of use.

8.1.2.5 Volatile acids (e.g., hydrochloric (HCl), hydrofluoric, nitric, and acetic) yield corresponding gases and vapors, especially when heated.

8.1.2.5.1 The reaction of hydrochloric and nitric acids in aqua regia yields nitrosyl chloride (NOCl) vapor and elemental chlorine (Cl<sub>2</sub>) gas.

8.1.2.5.2 When sulfuric acid is heated, it yields both sulfuric acid vapor (H<sub>2</sub>SO<sub>4</sub>) and sulfur trioxide (SO<sub>3</sub>).

8.1.2.5.3 Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) is not significantly volatile, but spray may be carried over into the exhaust system by mechanical entrainment.

8.1.2.5.4 Under appropriate conditions, spray droplets of any of the chemicals used may be mechanically entrained into the ventilation air exhausted from the various processes.

8.1.3 Design and selection of scrubbing equipment for the acid exhaust systems must take into account both

the chemical and physical properties of the materials being emitted.

8.1.4 The principles involved in collection of particulate matter (including liquid aerosols) are entirely different from those determining absorption of gases.

#### 8.1.5 Current Practice

8.1.5.1 Exhausts carrying acid compounds comprise the largest volume of air discharged from a typical semiconductor manufacturing plant.

8.1.5.2 The current practice is to connect most or all of the corrosive sources to a central building exhaust system and to discharge the combined gas streams to one or more large wet scrubbers.

8.1.5.2.1 A variety of scrubber types might be used, but the two main types are cross-flow and counter-current scrubbers equipped with packing material of some type.

8.1.5.3 The scrubbers usually are fed with a stream of water (potable, recycled, etc.).

8.1.5.4 A side stream of water is bled from the scrubber to a wastewater line.

8.1.5.5 It is futile to discharge organic solvents to water scrubbers even if the organic solvents are, to some degree, soluble in water.

8.1.5.5.1 Any dissolved organic solvent will eventually be desorbed from the water and will escape to the atmosphere, or be discharged as wastewater. Additionally, these organics could serve to propagate biological growth in the scrubber.

8.1.5.6 Exhausts from process tools are frequently discharged to the appropriate building exhaust systems, either with or without point-of-use abatement.

8.1.5.7 Arsine and phosphine, which have only slight solubility in water, pass through the water scrubbers so that the effect of discharging these gases into the building scrubber system is limited to dilution. Fluorine and chlorine also have limited solubility in water and may pass through water scrubbers without sufficient abatement. POU abatement devices should be considered for these types of gases.

8.1.5.8 Preliminary oxidation of these and numerous other process gases results in formation of fine fumes and other aerosols that will be collected with only low efficiencies by the typical large, low-energy wet scrubbers.

8.1.5.9 If ammonia is discharged into a system that also carries hydrogen chloride, hydrogen fluoride, nitric acid, or sulfuric acid; it will react, in the vapor phase, to

form sub-micron solid aerosols of ammonium chloride, ammonium fluoride, ammonium nitrate, or ammonium sulfate, respectively.

8.1.5.10 Any exhausts carrying ammonia should be discharged to a separate scrubber or a point-of-use ammonia abatement used at the process discharge.

#### 8.1.6 *Chemistry of Pollutants*

8.1.6.1 Effective abatement of any compound requires careful consideration of the chemistries and physical states of the specific compounds being abated.

8.1.6.2 The packed-bed scrubbers commonly employed should generally be adequate for absorption of readily soluble gases, if appropriate operating conditions are also maintained.

8.1.6.3 Misuse can negate the potential performance of the scrubbers.

8.1.6.4 As scrubbers are low-energy devices (pressure drops on the order of 2.5 to 10 cm (1-4 inches) of water, they are incapable of effectively collecting fine aerosols such as the ammonium salts mentioned above.

8.1.6.5 The most economic design approach, therefore, is to arrange the exhaust system and use POU abatement devices to avoid formation of such aerosols, where possible.

8.1.6.6 The practice of using only recirculated water in the scrubber, to absorb acid vapors and to neutralize the water bleed stream should be reviewed to ensure that scrubber efficiency is not affected.

8.1.6.7 Acid gases, such as hydrogen chloride and hydrogen fluoride, are readily soluble in water. The partial pressure of acid gases in equilibrium with the solution, which increases with increasing solute concentration, reduces the absorption of additional gas. Reduction in absorption can be minimized by increasing the rate of water blow-down (i.e., using more fresh water make-up).

8.1.6.8 However, increasing the rate proportionately increases the consumption of water and the volume of water that must be treated before discharge as wastewater.

8.1.6.9 The introduction of an alkaline solution (e.g., sodium hydroxide) in the scrubber can assist in removing compounds from the gas stream if the use of water alone does not provide adequate removal efficiencies. The use of additives in the scrubber can, in itself, create safety and environmental concerns.

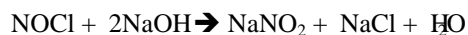
8.1.6.10 Some of the neutralized absorbent must be bled off from the recirculated stream to prevent the brine concentration from reaching an excessively high

level, but water consumption and the volume of waste can still be reduced by alkaline solution.

8.1.6.11 Aqua regia is a reactive mixture of nitric and hydrochloric acids. The reaction produces nitrosyl chloride and elemental chlorine:



8.1.6.12 The nitrosyl chloride vapor is readily decomposed by absorption in an alkaline solution:



8.1.6.13 Elemental chlorine has only a limited solubility in water, and an alkaline solution (pH > 10) must be used to attain adequate absorption efficiencies:



8.1.6.14 During the initial reaction of the nitric and hydrochloric acids, hydrogen chloride gas and nitric acid vapors are volatilized along with the nitrosyl chloride and chlorine.

8.1.6.15 Nitrogen dioxide may also be produced by decomposition of nitric acid.

8.1.6.16 All these compounds can be absorbed, to some degree, in an alkaline solution.

8.1.6.17 If a pollutant is in a particulate form (solid or liquid), its collection is only slightly affected by its chemical nature or solubility.

8.1.6.18 The dominant factor determining particulate collectability is particle size.

8.1.6.19 Abatement of particulate compounds requires a high-energy method (usually creating an extremely high pressure-drop across the abatement device) which can be costly in both energy and abatement device size requirements.

#### 8.1.7 *Scrubber Design and Selection*

8.1.7.1 A variety of commercially available scrubbers are applicable to absorption of readily soluble gases.

8.1.7.2 These include packed-bed counter-current, co-current, and horizontal cross-flow scrubbers, horizontal spray chambers, vertical spray towers, and gas-atomizing scrubbers such as the venturi scrubber.

8.1.7.3 To some extent, the choice of scrubber type is a matter of preference, as it is usually possible to obtain equivalent performance on the soluble gases with different types of devices; however, more packing or higher liquid loading may be required on some devices.

8.1.8 The choice is likely to be limited by a consideration of practical or economic factors such as cost, available space, and weight (e.g., it is possible to

reduce the size of the scrubber, usually at the cost of increased energy consumption).

NOTE 5: See Appendix 1 for absorber design criteria.

## 8.2 Group 2 — Acid Aerosols

8.2.1 Part of acid emissions may be in the form of mists (fine droplets or aerosols) rather than gases. These may be composed of relatively non-volatile acids such as phosphoric and sulfuric acids.

8.2.1.1 The mechanisms of removal of particulate matter (including liquid aerosols) from gas streams are entirely different from those involved in the absorption of gases (See Sections 13.1 through 13.3).

8.2.1.2 The critical factor in the collection process is the particle size of the mist. If the mist is relatively coarse, as apparently is the case in most instances, the same types of scrubbers that are used for gas absorption should collect the mists with adequate efficiency.

8.2.1.3 If a mist having a particle size in the micrometer or sub-micrometer range is encountered, it will be necessary to use a high-energy scrubber or a scrubber with a high-pressure-drop, sub-micron filter to obtain adequate collection efficiency.

8.2.1.4 Whenever such a fine mist or other aerosol is encountered, the gas stream from the source should be abated at point-of-use or separately from the other exhaust gas streams in the plant so as to minimize energy costs.

8.2.1.5 Abatement systems utilizing a scrubber with a high-pressure-drop, sub-micron filter are being used in the semiconductor industry to reduce emissions from aqua-regia processes, hot nitric baths and spray etchers using nitric acid (entrained in exhaust).

8.2.2 It has been determined experimentally that the collection efficiency of a scrubber on a given mist or dust is a function of the energy consumed in the exposure of the particle to the liquid in the scrubbing process.

8.2.2.1 The relationship between energy consumption and efficiency is little affected by the geometry or size of the scrubber or by the method by which the energy is applied to making contact between the gas and the liquid (See Sections 13.1 through 13.3).

8.2.2.2 The energy consumption required to attain a given efficiency increases with a decrease in the size of the aerosol (e.g., mist or dust). In most of the scrubbers used in the semiconductor industry, the energy consumed is drawn from the gas stream in the form of pressure drop.

8.2.3 The energy/efficiency relationship provides a convenient and practical method for particle and

aerosol scrubber design. It is essentially independent of the size of the scrubber, at least down to a very small size (perhaps 150 to 300 L/min (5–10 ft<sup>3</sup>/min) capacity, and possibly even smaller). Hence, a small pilot unit can be used to determine performance on an actual plant exhaust stream.

8.2.3.1 Tests can also be made under laboratory conditions, using synthetic aerosols generated for the purpose.

8.2.3.2 The major problem in using synthetic aerosols is in replicating the aerosols actually encountered in practice.

8.2.3.3 The pilot plant scrubber should be tested over a range of pressure drops to give a well-defined pressure drop/efficiency correlation.

## 8.3 Group 3 — Ammonia

8.3.1 Ammonia gas (NH<sub>3</sub>) can be either a process gas or evolved from ammonium hydroxide used in wet chemical cleaning of wafers. As previously discussed, ammonia (NH<sub>3</sub>) exhausted through the acid system will react with the acids present producing an ammonium salt aerosol that is not easily abated.

8.3.1.1 Packed-bed scrubbers are not very efficient at removing sub-micron ammonium halide aerosols, even though they are water-soluble.

8.3.2 Wet scrubber technologies, as listed above, are suitable for removal of ammonia provided they are operated and maintained at low pH (e.g., 3–5).

8.3.2.1 This is normally achieved by the use of sulfuric acid dosing.

8.3.3 Processes that emit both ammonia and acid gases (such as nitride deposition) should be fitted with a point-of-use ammonia scrubber, prior to being exhausted to the acid exhaust.

## 8.4 Group 4 — Volatile Organic Compounds (VOCs)

8.4.1 VOCs can represent a large proportion of exhausted air streams within typical semiconductor operations. The pollutants are produced from processes such as solvent cleaning, and photoresist application and stripping. Such processes typically contribute flammable, hazardous, and/or environmentally harmful compounds (e.g., VOCs, which participate in ozone formation in the atmosphere) to the exhausted air stream.

8.4.2 There are three primary abatement technologies used at end of pipe for this category: adsorption, recovery and oxidation. Adsorption generally uses hydrophobic zeolite or activated carbon. The oxidation process usually is thermal or may be catalytic.

#### 8.4.3 *Emission Sources and Chemicals Emitted*

8.4.3.1 VOCs usually have high vapor pressures and can be hazardous, flammable, or may tend to form photochemical smog. Processes using them (such as solvent cleaning, photoresist application, vapor degreasing and photoresist stripping) are carried out under local exhaust hoods or in exhausted enclosures.

8.4.3.2 Chemicals emitted may include: acetone, ethyl benzene, ethyl lactate, hexamethyldisilazane (HMDS), isopropyl alcohol, methanol, methyl ethyl ketone (MEK), n-butyl acetate, n-methyl-2-pyrrolidone (NMP), petroleum distillates (VM&P naphtha), propylene glycol monomethyl ether acetate (PGMEA) and xylenes.

8.4.3.3 The chemical species emitted are typically vapors of the specific VOCs being used. These vapors are entrained in large volume air streams and are thus diluted to low levels.

#### 8.4.4 *Current Practices*

8.4.4.1 New regulations and proposed legislation, being implemented worldwide, require significant reductions in total VOC emissions (often measured in terms of mass discharged).

8.4.4.2 Removal of VOCs from exhaust streams can be accomplished by adsorption, (e.g., fluidized bed and concentrator systems), oxidation, or a combination of adsorption and oxidation systems. Adsorption technologies, with subsequent desorption and condensation, can be used to recover VOCs as a liquid.

8.4.4.3 End-of-pipe abatement is most commonly used for VOCs. Point-of-use systems, using the same technologies as end-of-pipe, are employed where exhaust facilities preclude use of an end-of-pipe unit, or as additional abatement to meet regulatory requirements.

NOTE 6: See Appendix 1 for VOC abatement design and selection criteria.

#### 8.5 *Group 5 — Pyrophoric Gases*

8.5.1 Where separate exhausts for pyrophoric gases have been installed, it has been practice to fit a large air dilution chamber at the end of pipe. The reasoning has been that this will ensure safety by dilution and result in oxidation of any residual pyrophoric gases. However, oxidation may not occur since research has shown that silane, once diluted to <1.5 % in nitrogen, does not undergo appreciable oxidation when subsequently mixed with air.

8.5.2 Due to the risks associated with ducting pyrophoric gases through a facility, it is strongly recommended that these gases be abated at point-of-

use. This avoids the need for a separate pyrophoric gas exhaust. If POU abatement is provided, the discharge from the POU abatement system can be directed to the acid or general exhaust system.

NOTE 7: See Appendix 2 for POU abatement of pyrophoric gases.

8.6 *Group 6* — See section 10 for Emergency Release Discharge Exhaust provisions.

#### 8.7 *Group-7 — Special/Direct*

8.7.1 Recovery of specific gases such as hydrogen (H<sub>2</sub>) may be necessary to reduce the massive risk of duct fire when large quantities of flammable gases are discharged.

8.7.2 PFC reclamation maybe required by environmental permits in some jurisdictions.

8.7.3 Abatement of Oxides of Nitrogen (NO<sub>x</sub>) may be necessary to meet environmental regulations in some jurisdictions.

#### 8.8 *Group 8 — General Exhaust*

8.8.1 This exhaust group is used to handle exhaust emissions that include heat and excessive nitrogen flow.

8.8.2 Heat exhaust is used to reduce the heat load on the balance of the building.

8.8.3 Exhaust of large volumes of nitrogen or other asphyxiant gas is used to keep potential asphyxiation hazards away from people.

8.8.4 Discharge from general exhaust should be to a location where the heat or reduced oxygen will not be a hazard to personnel.

8.8.5 General exhaust can sometimes be used for discharge of post-treatment emissions from POU abatement systems.

8.8.5.1 In such a case, the discharge permit for the POU abatement system should be consulted for proper management.

#### 8.9 *Group 9 — Highly Toxic Gases*

8.9.1 Gases such as arsine, phosphine, diborane, germane, etc., can be treated in a variety of ways, depending upon their concentration and their byproducts (e.g., gas reactor columns, adsorption systems).

8.9.2 These gases would not likely be allowed in a burn system (even though many of them might be pyrophoric) because they could release hydrides from the air intake.

8.9.3 These gases may be prohibited, in some jurisdictions, from being discharged with no other treatment than dilution.

8.9.4 Highly toxic gases have also been known to create problems in a thermal oxidation system, because of the residue they leave behind and the periodic need for personnel to clean oxidation systems.

8.9.5 Selectors of systems for discharge of highly toxic gases should be careful to meet all regulatory requirements and should be conscious of the discharge location for the emission residue.

## **9 Point-of-Use (POU) Abatement Technologies**

9.1 The selection of a suitable POU abatement device is dependent on the process exhaust gases to be abated.

9.2 To determine a valid abatement method, identify the exhaust composition from the process as well as the input process gases in order to select a suitable abatement technology.

9.3 The choice may also depend on other factors (e.g., whether removal of PFC gases is required or not).

9.4 The location of a POU abatement system in the process exhaust train must be reviewed for pressure drop, condensation, particles, moisture backstreaming, etc.

9.5 Many types of POU abatement devices are available and they can broadly be divided into six types of technologies listed below.

1. Wet scrubbing systems,
2. Oxidation systems,
3. Cold bed systems (adsorbers/ chemisorbers),
4. Hot chemical bed systems,
5. Reactor systems (e.g., plasma, microwave),
6. Traps/filters/cyclones/precipitators,

NOTE 8: See Appendix 2 for explanations of each technology type.

9.6 Units may employ one or more of these technologies, depending on the application.

9.7 Where a technology using an exhaustible cartridge/canister is employed, consideration must be given to adequate means of detecting bed exhaustion and to proper disposal techniques.

## **10 Provisions For Emergency Release**

10.1 Routine releases, from hazardous gas cylinders and sources are usually minimal (e.g., from gas line purges).

10.2 Emergency or accidental releases, from gas cylinders in use, may be controlled through mechanical systems that lower the frequency, time and rate of releases.

10.3 Enclosing gas cylinders and non-welded mechanical fittings in exhausted gas cabinets that protect the systems from damage can minimize the frequency of accidental releases.

10.3.1 Proper design and installation of gas delivery and purge systems can reduce the likelihood of severe leaks.

10.4 The duration of accidental releases can be lessened by activation of fail-closed valves or gas cylinder closure devices activated by monitoring and/or alarm systems.

10.5 Equipping gas cylinders with restrictive flow orifices can lower the rate of accidental release. SEMI S5 - Safety Guideline for Flow Limiting Devices provides guidance on the use of restrictive flow devices.

10.6 Exhaust of the gas cabinets should be designed to contain sudden unanticipated releases of gas from cylinders and piping.

10.6.1 These exhausts are typically used to manage gas cabinets and other very high volume exhaust flow systems that will only occasionally have emissions present.

10.6.2 Some jurisdictions do not allow for discharge without treatment of emergency release discharges.

10.7 Each system selector should research their own regulations before determining the proper route and method for discharging and treating emergency releases.

10.8 Accidental releases from gas cylinders in use may be controlled through mechanical systems that lower the concentration exhausted into the environment. In some jurisdictions there are requirements (e.g., Uniform Fire Code, Article 80) to lower the concentration of accidental release, in exhaust to the environment, below a threshold (such as one-half (1/2) of Immediately Dangerous to Life and Health (IDLH) levels).

10.8.1 Dilution air, in excess of otherwise required gas cabinet exhaust, can be used to reduce the concentration of gas accidentally released into the exhaust, except where a jurisdiction specifically prohibits this practice.

10.8.2 As another method to reduce the concentration of gas in the atmospheric discharge during accidental releases, abatement devices serving the gas cabinet

exhaust can be used instead of, or in addition to, dilution air.

10.8.2.1 Accidental release abatement devices may be any of the types described in this guide.

10.8.2.2 In some applications, gas cabinet exhaust is diverted to an abatement device only upon detection of an accidental release.

10.8.2.3 When designing diversion systems, the ability of the abatement device to operate after extended periods of disuse must be considered.

## 11 Summary Table

11.1 See Table 1 for suggestions about using POU and end-of-pipe abatement systems for different types of processes, materials, and exhaust systems.

## 12 Alternative Approaches

12.1 This guide is primarily concerned with handling gas effluents that arise from semiconductor processing. Capture and abatement is not the only approach to avoid release of hazardous materials. If possible, use of the hazardous materials should be avoided. In theory the following approach should be used:

12.1.1 Replace the hazardous material with one that is non-hazardous or less hazardous.

12.1.2 If replacement is not possible, use less of the material.

12.1.3 If possible, totally enclose the system so all material is recovered and reused.

12.1.4 If none of the above can be achieved, fit effective abatement devices.

## 13 Related Documents

NOTE 9: Unless otherwise indicated, all documents cited shall be the latest published versions.

### 13.1 SEMATECH Documents<sup>3</sup>

TT97093364A-XFR — Point-of-Use (POU) Control Systems for Semiconductor Process Emissions (ESH003), October 30, 1997, J. Michael Sherer, P.E., Motorola (available online from [www.semtech.org/public/docubase/summary/3364AXFR.htm](http://www.semtech.org/public/docubase/summary/3364AXFR.htm))

### 13.2 FM Global Documents<sup>4</sup>

3 SEMATECH/International SEMATECH, 2706 Montopolis Drive, Austin, Texas 78741-6499

4 FM Global Corporation, 1151 Boston-Providence Turnpike, Norwood, MA 02062 or the FM Global Web Site at: [www.fnglobal.com](http://www.fnglobal.com)

Property Loss Prevention Sheet 7-78 — Industrial Exhaust Systems

### 13.3 Other Documents

Design and Selection of Spray/Mist Elimination Equipment<sup>5</sup>

Toxicological Investigation in the Semiconductor Industry<sup>6</sup>

Reactions of Exhaust Deposits from Silicon Deposition Tools<sup>7</sup>

Treatment of Organochlorines from Plasma Etch Processing<sup>8</sup>

Toxicological Hazards of Plasma Etch Waste Products<sup>9</sup>  
Packed Column Internals<sup>10</sup>

### 13.4 I300I/International Sematech<sup>11</sup>

Emissions Characterization 2.4b — [www.I300I.org](http://www.I300I.org), Zero Impact Process Team.

5 Chemical Engineering Volume 91, No. 21, 82–89 (October 15, 1984), Holmes, T. L.; and Chen, G. K.

6 Toxicology & Industrial Health 8 (141) 1992 by Bauer, et. al.

7 Semiconductor Safety Association Conference, Orlando, April 1997 by Creighton S.; Plaster, M.; and Nicholson, T.

8 Semiconductor Safety Technology, March 1995 by Baker, D.; Smith, J.; and Mawle, P.

9 Semiconductor Safety Technology, July 1996 by Bauer, F.; Wolff, I.; and Schmidt, R.

10 Chemical Engineering, Volume 91, No. 5, pp 40–51 March 5, 1984 by Chen, G. K.

11 I300I/International Sematech, 2706 Montopolis Drive, Austin, Texas 78741-6499



**Table 1 Exhaust System Selection Table**

<i>Process</i>	<i>Gases/Chemicals</i>	<i>Reason for POU Abatement</i>	<i>POU Abatement Present</i>	<i>End-of-pipe Exhaust System Type</i>
Solvent/strippers	VOC		Yes	General
			No	VOC
Vapor prime	HMDS		No	Acid or ammonia
Acid baths	HCl/HF/Acetic		No	Acid
Acid baths	Aqua regia/hot nitric		No	Acid aerosols
			Yes	Acid
Ammonia baths	NH <sub>3</sub>		No	Ammonia
			Yes	Acid
Gas bottle purge	Corrosives		No	Acid
			Yes	Acid
	Ammonia		No	Ammonia
			Yes	Acid
	Hydrides/pyrophorics		Yes	General
Chemical dispensing			No	General
	Ammonia		No	Ammonia
	VOC		No	VOC
Chemical wafer thinning of raw wafers	Corrosives		No	Acid
	HNO <sub>3</sub> /NO <sub>x</sub>		Yes	Acid
			No	NOx/acid scrubber
EPI	H <sub>2</sub> /Hydrides/HCl	Fire and safety Solids blockage	Yes	Acid, H <sub>2</sub> reclaim, or general
Dry etching – metal	Cl and Br chemistries	Blockage/Corrosion	Yes	Acid
Dry etching – oxide/poly/etc.	Fluoride chemistry		No	Acid
LPCVD nitride	SiH <sub>2</sub> Cl <sub>2</sub> /NH <sub>3</sub>	Solids blockage Fire and safety	Yes	Acid
LPCVD poly/oxide	SiH <sub>4</sub> /fluoride chemistry	Fire and safety Solids blockage	Yes	Acid
PECVD – poly/oxide	SiH <sub>4</sub> /fluoride chemistry	Fire and safety Solids blockage	Yes	Acid
PECVD nitride and tungsten	SiH <sub>4</sub> /WF <sub>6</sub> /H <sub>2</sub>	Fire and safety Solids blockage	Yes	Acid
	SiH <sub>4</sub> /NH <sub>3</sub>	Fire and safety Solids blockage	Yes	Acid
Implant	Hydrides, BF <sub>3</sub>	Hazardous deposits	Yes	Acid
RTP	Ammonia		No	Ammonia
			Yes	General

NOTE T1: POU Abatement may be used for the reduction of PFCs and by-products from the process.

## APPENDIX 1

### END-OF-PIPE ABATEMENT DESIGN

NOTE: The material in this appendix is an official part of SEMI F5 and was approved by full letter ballot procedures on August 27, 2001.

#### A1-1 Referenced Documents

A1-1.1 Gas-Liquid Reactions<sup>12</sup>

A1-1.2 Design and Selection of Spray/Mist Elimination Equipment<sup>13</sup>

A1-1.3 Absorption, Distillation, and Cooling Towers<sup>14</sup>

A1-1.4 Perry's Chemical Engineers' Handbook<sup>15</sup>

A1-1.5 Absorption and Extraction<sup>16</sup>

A1-1.6 Mass Transfer<sup>17</sup>

A1-1.7 Mass-Transfer in Co-current Gas-Liquid Flow<sup>18</sup>

A1-1.8 Mass-Transfer Operations<sup>19</sup>

#### A1-2 Principles

A1-2.1 Principles of gas absorption and methods of design of gas absorption equipment are treated extensively in chemical engineering literature (See Sections A1-1.1–A1-1.8). The sources cited present rigorous methods for design.

A1-2.2 Practical design of scrubbers is at least semi-empirical; and in the case of air pollution abatement systems, design is often directly based on experimental data.

A1-2.2.1 Consequently, designs should be based on mass-transfer coefficients that have been determined experimentally under, as nearly as possible, the conditions of expected scrubber operation.

A1-2.2.2 It should be noted that the typical concentrations of the existing empirical data base is several orders of magnitude greater than the typical concentrations of corrosives exiting a semiconductor

plant (before end-of-pipe scrubbers), therefore the efficiencies in these data bases may not be representative of low concentrations.

A1-2.2.3 Attempts at developing the empirical database at these low concentrations suggest that actual removal efficiencies are lower than those predicted by most scrubber design texts.

A1-2.2.4 As a result, new scrubbers may need to have additional packing to ensure theoretical removal efficiencies are achieved.

A1-2.3 Rigorous custom design of a scrubber for a particular service requires full knowledge of the conditions that are to be encountered as well as of the performance that is to be attained.

A1-2.3.1 Gas flow rates should normally be known fairly accurately — at least when the plant is originally designed.

A1-2.3.2 Concentrations of acid and caustic gases will need to be estimated.

A1-2.3.3 Conditions to be met may be changed several times over the probable life of the scrubbing equipment, as changes are made in the semiconductor fabrication processes and the exhaust system.

A1-2.3.4 Exhaust flow may also be increased, to accommodate equipment additions, thus raising the possibility the scrubber may become overloaded.

A1-2.4 Studies by SEMATECH indicate that the concentration of corrosives in the exhaust entering the end-of-pipe scrubber(s) range from <1 ppmv to as high as 10 ppmv. Additionally, the typical scrubber with 99+ % theoretical removal efficiency was actually achieving removal efficiencies ranging from 80%–95%.

A1-2.5 Scrubbers being applied to corrosive pollutants are usually standardized units assembled by a number of vendor companies. These units are designed to handle specified ranges of gas flow, but in other respects are not designed but simply built. If they are packed-bed scrubbers, they are usually fitted with standard depths of packing. The liquid flow rates can be varied within limits; but otherwise, the performance potentials are fixed unless there are provisions for changing the depth of packing and perhaps the kind of packing. Once the system is installed, such changes to increase the efficiency will probably increase the

12 McGraw-Hill, New York (1970), Danckwerts, P.V.

13 Chemical Engineering Volume 91, No. 21, pp 82-89 (October 15, 1984), Holmes, T. L.; and Chen, G. K.

14 Longmans, Green, London (1961), Norman, W. S.

15 6th Edition, McGraw-Hill; New York (1984), Perry, R. H.; and Green, D.

16 2nd Edition; McGraw-Hill, New York (1952), Sherwood, T. K.; and Pigford, R. L.

17 McGraw-Hill, New York (1975), Sherwood, T.K.; Pigford, R. L.; and Wilke, C. R.

18 Chemical Engineering Sciences Volume 33, No. 12, pp1675-1680 (1978), Shilimkan, R. V. and Stepanek, J. B.

19 3rd Edition; McGraw-Hill, New York (1980), Treybal, R.E.

pressure drop of the scrubber, which will require adding to the fan capacity.

A1-2.6 To ensure, in advance, that the scrubber will have adequate removal efficiency, performance tests can be conducted. Measurements should be made of the contaminant concentrations in typical plant exhaust gas streams, and the resulting data should be used to set conservative performance requirements for the scrubber. Performance tests on the scrubber should be conducted prior to exposing the scrubber to the semiconductor plant exhaust, preferably at the vendor's facility. Performance tests should identify the critical operating variables and optimize the settings to achieve optimal removal efficiencies at the lowest cost.

A1-2.6.1 Onsite performance tests may be preferable, when qualifying abatement systems, so that accurate process conditions can be produced. Permit exemptions may be obtained for the time-period of system qualification.

A1-2.7 Design and construction of scrubbers are simplified by two factors: the exhaust gas streams are at essentially ambient temperature, and they carry few insoluble solids. The low temperature allows use of corrosion-resistant plastic construction. The absence of significant solids avoids blockages in the scrubbers, which favors use of efficient gas-liquid contactors such as packed beds.

A1-2.8 Two of the most favored scrubber types are the counter-current packed tower and the horizontal cross-flow scrubber. In some instances, the choice between these devices may be determined by plant layout. As usually constructed, these two scrubber types have low to moderate gas pressure drops and are primarily suited to absorption of gases. They will also collect the coarser mists and sprays but are unsuited to collection of fine mists and fumes having particle sizes under 2 to 3 micrometers.

A1-2.9 In principle, the counter-current tower offers the highest efficiency with the least consumption of absorbent liquid. However, if untreated water is recirculated through the tower, as is commonly done in semiconductor plants, the advantages of the countercurrent operation are lost. On the other hand, if the absorbed gas is reacted with an appropriate reagent in the water, essentially the same performance can be obtained with either the countercurrent or co-current operation (see Section A1-1.7). In co-current operation, it is possible to use higher gas velocities to obtain higher mass transfer rates so that the scrubber can be made smaller. Gas pressure drops will also be increased, but this may be desirable if it is necessary to collect a fine aerosol as well as absorb a gas.

A1-2.10 Horizontal cross-flow packed-bed scrubbers may have advantages where the installation imposes restrictions on height or other similar layout problems. However, in other respects, they do not offer fundamental advantages over the packed tower scrubbers.

A1-2.11 Under special circumstances, other types of scrubbers may be appropriate for collection of aerosols. High-energy scrubbers, of the venturi type, or scrubbers with high-pressure drop, sub-micron filters (e.g., fiber bed) are appropriate where fine aerosols must be collected. Wherever possible, measures should be taken to avoid formation of such aerosols; but when they are unavoidably produced, the source should be ventilated through a separate scrubbing system of appropriate design.

A1-2.12 Packing in scrubbers provides a large interfacial area between the liquid and the gas, and also induces fluid turbulence to promote mass transfer. Packing is commonly placed in scrubbers in a random manner. Raschig and Pall rings as well as Berl saddles were common early packing materials used. In recent years, numerous proprietary packing designs have been introduced, with the objective of providing increased mass-transfer efficiency with reduced gas pressure drop. The validity of claims for superior performance has been established by the SEMATECH experiments in Section 13.1.

A1-2.13 Numerous precautions are required when installing and operating packed-bed scrubbers. It is essential to attain an even distribution of liquid over the packing; channeling of liquid or gas flow results in loss of removal efficiency. Inadequate liquid distribution can result in a reduction of the removal efficiency to as little as one seventh of theoretical (See Referenced Document A1-1.9). The predominant liquid distribution system employed is spray nozzle style. This style is inexpensive, however, it is difficult to maintain and tends to entrain more water than weir-style. Weir-style liquid distribution systems are more expensive. However, they require less preventive maintenance, provide equal liquid distribution over a wide range of flow rates, and are less prone to liquid carry-over. Precautions are treated at length in previously cited literature.

A1-2.14 A critical aspect of any scrubber design is the separation of entrainment. Carry-over of spray from a scrubber can produce maintenance issues and mist emissions. Failure to provide adequate entrainment separation has been one of the most common problems in scrubber installations. The predominant mist separator employed is a mesh style. Mesh style separators are effective on the smaller water droplets associated with spray nozzles; however, maintenance

due to solids and/or biological growth is required. The chevron style is typically used in conjunction with a weir, as they are not very effective on the smaller droplet associated with spray nozzles; however they are very immune to plugging from solids and/or biological growth. The entrainment separators are discussed in Sections A1-1.3 and A1-1.5.

A1-2.15 Chlorine, fluorine and possibly other soluble gases can be entrained in scrubber wastewater, and released in facilities drains/vents. Materials such as fluorine are incompatible with many plastics and can damage drains/ vents resulting in leak, odors, and facilities interruptions.

### **A1-3 VOC Abatement Equipment Design and Selection**

#### *A1-3.1 Design Criteria for Adsorption Systems*

A1-3.1.1 Adequate contact time must be achieved between adsorption media and VOC-laden exhaust stream. The adsorption matrix must be sized so that adequate residence time within the media is maintained. Also, the exhaust stream flow must be distributed uniformly over the adsorption matrix.

A1-3.1.2 Pressure drop across the adsorption matrix should be minimized to reduce energy consumption for air handling equipment.

A1-3.1.3 Materials of construction must be resistant to any corrosive action by the VOC' s or by-products formed during adsorption or adsorbent matrix regeneration.

A1-3.1.4 Hydrophobic zeolites and activated carbon in fixed beds, fluid beds or rotating wheels are being used for adsorption.

A1-3.1.5 Adsorption materials have a finite capacity for VOC adsorption and must be regenerated when their capacity is exhausted.

A1-3.1.6 Monitoring of the exhaust stream as it exits the adsorption bed may be necessary to determine when regeneration or replacement of the adsorption medium is needed.

A1-3.1.7 Pretreatment of the VOC-laden exhaust stream for removal of entrained particles or liquids may be required to prevent plugging of the adsorption unit.

A1-3.1.8 Regeneration is accomplished by flushing the adsorption bed with a carrier, typically steam, hot air or hot nitrogen.

A1-3.1.8.1 Desorbed VOC' s must be recovered or destroyed.

A1-3.1.8.2 Current practices for VOC recovery requires condensation or distillation.

A1-3.1.8.3 Current practices for VOC destruction is usually oxidation.

#### *A1-3.2 Design Criteria for Oxidation Systems*

A1-3.2.1 Adequate residence time, mixing with air and temperature, must be provided for high efficiency destruction of the VOC' s.

A1-3.2.2 Pretreatment of the VOC-laden exhaust stream for removal of entrained particles or liquids may be required to prevent plugging of the oxidation unit.

A1-3.2.3 Downstream abatement of the oxidation unit exhaust will be required if corrosive combustion products [such as hydrogen chloride (HCl) from chlorinated vapors] or particles [such as silicon dioxide (SiO<sub>2</sub>) from hexamethyldisilazane (HMDS)] are present.

A1-3.2.4 Concentrations of the incoming VOC' s should be limited, especially when using a concentrator. Typical incoming VOC concentrations should not exceed 25% of LEL.

A1-3.2.5 Energy costs for heating VOC-laden exhaust streams must be considered. Straight thermal oxidizers are costly to use with high-volume, low VOC concentration exhaust streams. Therefore, a concentration process followed by recuperative or regenerative oxidation offers a more cost-effective solution.

A1-3.2.5.1 Catalytic oxidation may also offer cost savings; however, the composition of incoming compounds must not poison the catalyst.

A1-3.2.5.2 A regenerative thermal oxidizer (without a preceding concentrator) may offer cost savings in certain applications.

A1-3.2.6 Hybrid systems (combinations of adsorption and oxidation systems) may provide a cost-effective approach for abating dilute concentrations of VOCs in exhaust streams by combining adsorption beds with recuperative/regenerative oxidation, to destroy the VOCs.

A1-3.2.6.1 All design considerations for adsorption systems and oxidation systems are relevant for any of the multitude of possible hybrid systems.

A1-3.2.6.2 Levels of oxidation by-products must also be considered such as oxides of nitrogen (NO<sub>x</sub>), carbon monoxide (CO) and products of incomplete combustion (PICs).

A1-3.2.7 Potential problem areas to be reviewed in any equipment selection procedure:

A1-3.2.7.1 *Halogenated Materials* — Additional care must be exercised with chlorinated compounds, which



can be subject to stricter regulatory requirements. Ensure that any oxidation unit does not create new and more hazardous compounds, such as dioxins.

*A1-3.2.7.2 Sulfur and Hexamethyldisilazane (HMDS)*

— Both have the potential to poison many catalysts, and hexamethyldisilazane (HMDS) will oxidize to form silicon dioxide, which can result in particle build up in heat exchangers and oxidizers.

*A1-3.2.7.3 Moisture Levels* — Moisture can significantly affect adsorption capability.

## APPENDIX 2

### POU ABATEMENT TECHNOLOGY TYPES

NOTE: The material in this appendix is an official part of SEMI F5 and was approved by full letter ballot procedures on August 27, 2001.

#### A2-1 POU Abatement Technology Types:

1. Wet scrubbing systems
2. Oxidation systems
3. Cold bed systems (adsorbers/ chemisorbers)
4. Hot chemical bed systems
5. Reactor systems (e.g., plasma, microwave)
6. Traps/filters/cyclones/precipitators

#### A2-2 Wet Scrubbing Systems

##### A2-2.1 Water Scrubber

**A2-2.1.1 Principle of Operation** — Exhaust gas is passed through an enclosed space into which water is sprayed. The desired result is that gases will dissolve in or react with the water. A large contact area between gas and water is required to maximize the dissolution or reaction. Numerous designs exist to achieve this. The “packed-bed” scrubber is filled with open structure objects (e.g., hollow balls) that are fully wetted by the water spray. The gas passes over this large wet surface area. If the water spray is in the opposite direction to the gas path the design is “counter current”; if in the same direction, it is “co-current”; and if at 90° it is “cross flow”. Other designs to maximize gas/water contact use meshes, atomized sprays, or multi chamber systems that reverse the gas direction several times.

**A2-2.1.2 Capacity** — Total gas flow is determined by the size of the unit.

**A2-2.1.3 Efficiency** — Efficiency is determined by the solubility of the gas in water and retention time in the unit as well as the given mass transfer from gas to liquid phase. Acid gases are best removed; many other process gases may not be removed. If gas does dissolve in water then effectiveness of unit will be determined by design input gas flow and inlet compound concentrations.

**A2-2.1.4 Limitations** — Water scrubbers are only suitable for water-soluble gases. Many process gases (e.g., silicon compounds) will produce insoluble solids (silica) on contact with water resulting in solids forming that may block the water sprays, scrubber packing or gas inlet port.

NOTE 1: Compatibility of scrubber drains with the compounds being entrained in the liquid effluent should be considered.

##### A2-2.2 Water Scrubber with Energy Input (e.g., Venturi)

**A2-1.2.1 Principle of Operation** — Same as water scrubber but gas and water are forced to mix by the input of energy. Most commonly this is done by a venturi through which the gas and water pass. Other designs have used rapidly rotating blades in the enclosed scrubbing space. The “active” scrubbing area is often combined with a passive spray chamber or packed bed.

**A2-2.2.2 Capacity** — Energy enhanced water scrubbers have the same capacity as water scrubbers.

**A2-2.2.3 Efficiency** — Added energy improves efficiency over water scrubbers due to forced mixing, which improves efficiency of particulate removal.

**A2-2.2.4 Limitations** — The limitations are the same as for water scrubbers.

##### A2-2.3 Chemical Dosed Scrubber

**A2-2.3.1 Principle of Operation** — Chemical Dosed Scrubbers operate the same as a water scrubber but a chemical solution is added to the water. This increases the range of gases that can be removed, from those that dissolve in water to those that will react with the chosen chemical solution. The chemical medium is selected to react with the known input gases producing water-soluble salts. The two reaction systems most commonly employed are acid/base and reduction/oxidation (redox). Addition of solutions of sodium hydroxide (NaOH), potassium hydroxide (KOH) or sulfuric acid are frequently used to enhance effectiveness, and where chemical oxidation is needed hypochlorites, periodates, or peroxides are introduced.

**A2-2.3.2 Capacity** — Same as water scrubber.

**A2-2.3.3 Efficiency** — Same as water scrubber but increased efficiency by using chemical dosing. The number of gases, which can be removed, will also increase.

**A2-2.3.4 Limitations** — Even though a wider range of gases can be removed with this chemical dosing, it is applicable only to those emissions that can be water scrubbed. The potential for fouling by silica solids

remains. Potassium hydroxide (KOH) or (sodium hydroxide (NaOH) solutions have to be increased to approximately 5% to ensure dissolution of silicates. Chemical dosing adds another hazardous material and may result in liquid wastes that require treatment.

#### A2-2.4 *Chemical Dosed Scrubber with Energy Input (e.g., Venturi)*

A2-2.4.1 *Principle of Operation* — Same as Water Scrubber with Energy Input (e.g., Venturi) above, with the benefits of Chemical Dosed Scrubber.

### A2-3 Oxidation Systems

#### A2-3.1 *Burn Systems*

A2-3.1.1 *Principle of Operation* — Burn systems are designed for silane abatement by utilizing the pyrophoric properties of this gas. The process gas containing silane is injected into a steel container through which a stream of air is passing. The silane auto-ignites and oxidizes to form silica powder, which deposits in the system or is carried into the exhaust duct.

A2-3.1.2 *Capacity* — Governed by the build up of powders, small units may need cleaning out every few months, larger units will provide a year's capacity.

A2-3.1.3 *Efficiency* — When burn systems are correctly set up, nearly complete oxidation will occur, providing that the input silane concentration is greater than 1.5%.

A2-3.1.4 *Limitations* — Burn systems are suited only to silane (or other pyrophoric effluent) abatement. Not appropriate for mixed gas process. If silane is diluted to less than 1.5% in nitrogen before input no measurable oxidation will occur and the silane will pass directly to the exhaust.

#### A2-3.2 *Flame Oxidation*

A2-3.2.1 *Principle of Operation* — Oxidation of hydride gases by combustion. These systems use a chamber containing a fuel gas flame through which the process gas is passed. The resulting oxides from combustion pass into the exhaust and may be abated downstream.

A2-3.2.2 *Flow Capacity* — To calculate flow capacity, combine the total discharge flows that constitute input to the abatement device. Include pump dilution and purge quantities. .

A2-3.2.3 *Efficiency* — For simple burner designs, efficiency can vary widely as gas flow changes; specific units have been designed to ensure good mixing of fuel/air and gas; these can deliver efficiencies greater than 99.9%.

A2-3.2.4 *Limitations* — Suited only to processes where the input gases can be safely combusted. Design must ensure by-products are minimized (e.g., chlorinated compounds like phosgene/dioxins and oxides of nitrogen). Produces combustion byproducts of process gases, therefore, is often combined with a wet scrubber to remove by-products or other water-soluble gases.

#### A2-3.3 *Hot Chamber Oxidation*

A2-3.3.1 *Principle of Operation* — Exhaust gas is passed into an electrically heated chamber (typically 800° C) where it is mixed with air. The heated mixture oxidizes, more quickly than in a simple burn system, but without a flame from fuel oxidation to assist.

A2-3.3.2 *Flow Capacity* — To calculate flow capacity, combine the total discharge flows that constitute input to the abatement device. Include pump dilution and purge quantities.

A2-3.3.3 *Efficiency* — Hydride gases will be oxidized and removed to below the OEL.

A2-3.3.4 *Limitations* — On its own, hot chamber oxidation technology is suited only to hydrides or other gases that will readily oxidize at the temperature of the hot chamber. This method is often combined with a wet scrubber to remove oxidized by-products or other water-soluble gases.

#### A2-3.4 *Non-Flame Oxidation*

A2-3.4.1 *Principle of Operation* — Non-flame systems use an incandescent porous wall for combustion without a flame. Fuel and air are mixed and passed through the matrix of the porous wall, which operates at around 900° C. When process exhaust gases are passed into this zone, they are oxidized.

A2-3.4.2 *Flow Capacity* — To calculate flow capacity, combine the total discharge flows that constitute input to the abatement device. Include pump dilution and purge quantities.

A2-3.4.3 *Efficiency* — Hydride gases can be oxidized by a method similar to other combustion techniques. High efficiency oxidation is needed to oxidize more stable fluorinated compounds.

A2-3.4.4 *Limitations* — Non-flame oxidation produces oxidation by-products, and therefore must often be combined with a wet scrubber to remove particulate matter or water-soluble gases.

### A2-4 Cold Bed Systems (Adsorbers/Chemisorbers)

#### A2-4.1 *Adsorption*

A2-4.1.1 *Principle of Operation* — Exhaust gas is passed through a container (cylinder, canister, or drum)

filled with adsorbent granules. These are commonly based on activated charcoal. Some gases are physically adsorbed into the granules while the non-adsorbed gases pass through into the facility exhaust.

**A2-4.1.2 Flow Capacity and Removal Capacity** — Both capacities of these systems are a function of the container size (mass of granules) and surface area.

**A2-4.1.3 Efficiency** — For a single gas, efficiency can approach 100% until the bed capacity is reached. However, since the gas is only held physically it can be displaced by another compound, causing the efficiency to drop significantly.

**A2-4.1.4 Limitations** — Adsorption traps hazardous compounds and does not destroy them. Gases are adsorbed at different rates and the capacity of the bed for different gases will also vary. A more strongly adsorbed gas entering the bed after a less strongly adsorbed compound can displace the first gas releasing the first gas to the exhaust. The waste material must be kept sealed in the container and evaluated for hazards. Exposure to air, water, or heat can cause gases adsorbed in the material to be released.

#### **A2-4.2 Chemisorption**

**A2-4.2.1 Principle of Operation** — Exhaust gas is passed through a container of granules that consist of either:

- adsorbent granule coated with some reactive chemical;
- reactive porous medium e.g., soda lime; or
- a resin matrix coated with some reactive chemical.

**A2-4.2.1.1** Gases are adsorbed into the porous matrix and then chemically react either with the granules themselves or with the coating. These reactions are designed to convert the gas into a solid material (typically a salt) that remains attached to the granules.

**A2-4.2.1.2** Reactive chemicals are typically either bases (metal hydroxides) or oxidizers (e.g., permanganates, or metal oxides).

**A2-4.2.2 Flow Capacity and Removal Capacity** — Both capacities of these systems are a function of the container size (mass of granules) and surface area.

**A2-4.2.3 Efficiency** — Acid/Base and Redox reactions are most readily accomplished. Therefore, with acidic gases and hydride gases, efficiency can be high; with removal below OEL. With other gases, e.g., PFCs and halogenated carbon compounds, efficiency is often minimal or none.

**A2-4.2.4 Limitations** — There may be adsorption as well as chemisorption, so the spent material must be

evaluated for hazards, since gases can desorb from it. This is especially true for chlorine ( $\text{Cl}_2$ ) when the bed uses hydroxides. These will form OCl compounds that on exposure to air or moisture will regenerate chlorine.

**A2-4.2.4.1** Materials used for chemisorption can themselves be hazardous (e.g., heavy metals or caustic (KOH/NaOH)) therefore, unused canisters may be classified as hazardous items.

#### **A2-4.3 Adsorption with Subsequent Air Oxidation**

**A2-4.3.1 Principle of Operation** — These systems are designed and offered mainly for high flows of arsine ( $\text{AsH}_3$ ) and phosphine ( $\text{PH}_3$ ), used in the deposition of III-V materials (such as gallium arsenide (GaAs), indium phosphide (InP)). These gases are adsorbed into a carbon bed, into which air is subsequently fed at a controlled rate to oxidize the adsorbed hydride into oxides that will remain in the granules.

**A2-4.3.2 Flow Capacity and Removal Capacity** — Both capacities of these systems are a function of the container (drum) size (mass of granules) and surface area.

**A2-4.3.3 Efficiency** — Capable of removing hydrides to below OEL.

**A2-4.3.4 Limitations** — Hydride is adsorbed into the carbon bed, the oxidation of this is exothermic. When air is fed in to oxidize the hydride, it is possible to start combustion of the carbon bed, unless the flow rate is well controlled.

### **A2-5 Hot Chemical Bed Systems**

#### **A2-5.1 Hot Bed Reactors**

**A2-5.1.1 Principle of Operation** — Exhaust gases are passed through a steel container (cartridge/drum) that is filled with reactive material. The material is maintained at an elevated temperature, typically 250 to 550 degrees C. The gases chemically react with the fill material and are converted into inorganic salts that remain fused into the matrix of the fill material. There are versions with a single fill material that are specific to abatement of one gas, and versions with two or more zones of fill material that can abate a wide range of gas compounds.

**A2-5.1.2 Flow Capacity and Removal Capacity** — Both capacities of hot bed systems are a function of the cartridge size (mass of granules) and surface area.

**A2-5.1.3 Efficiency** — Efficiency of these systems is generally high, with gases removed to below OELs. Stable PFC gases are not removed by all systems.



A2-5.1.4 *Limitations* — Fill material can also react with oxygen or moisture so hot bed systems are not always suited to wet processes or where air is used.

#### A2-5.2 *Hot Catalytic Bed*

A2-5.2.1 *Principle of Operation* — Gas is passed into a steel cartridge, which is held at an elevated temperature, that contains granules of a catalyst. The target-input gas is catalytically converted to other gases [e.g., ammonia ( $\text{NH}_3$ ) to nitrogen ( $\text{N}_2$ ) and hydrogen ( $\text{H}_2$ ), and oxides of nitrogen ( $\text{NO}_x$ ) to nitrogen ( $\text{N}_2$ ) and oxygen ( $\text{O}_2$ )].

A2-5.2.2 *Removal Capacity* — In theory, if not poisoned, a catalyst bed should last for a long time. Units available are claimed to be able to operate for one-year before bed change.

A2-5.2.3 *Efficiency* — Efficiency of hot catalytic bed systems is normally high, with removal of the target gas to below OEL levels.

A2-5.2.4 *Limitations* — Hot catalytic bed units are designed to be gas specific. Catalysts can be sensitive to other compounds; so for complex gas mixtures in process exhausts they may not be suitable on their own.

#### A2-5.3 *Hot Reactor Beds with Gas Inputs*

A2-5.3.1 *Principle of Operation* — Exhaust gases are passed into a hot bed as in hot bed reactors above, but concurrently with the process gas another gas is introduced (e.g., air or steam). The gases then react together in the bed, either with each other or with the bed material. Hazardous gases are converted either to solid salts that remain fused into the bed matrix, or other gases [e.g., carbon dioxide ( $\text{CO}_2$ )] that pass on into the exhaust.

A2-5.3.2 *Flow Capacity and Removal Capacity* — Both capacities of these systems are a function of the container (cartridge) size (mass of granules) and surface area.

A2-5.3.3 *Efficiency* — Process gases [e.g., phosphine ( $\text{PH}_3$ ), carbon monoxide ( $\text{CO}$ )] are normally removed to below the OEL.

A2-5.3.4 *Limitations* — These systems are suited only to processes where the end products will be either stable solids or inert gases.

### A2-6 Reactor Systems (Plasma, Microwave, etc.)

A2-6.1 *Principle of Operation* — Gases are abated in the vacuum line (before or after the vacuum pump) by passing them through a reaction chamber, containing plasma, to enhance reaction of gases such as silane into a solid material. For high efficiency PFC conversion, it

is necessary to inhibit recombination by the addition of a material (e.g., moisture, or a hydrogen/oxygen mixture). Two types of reactors exist - large plasma volume/small surface area, in which a downstream particle trap is utilized; and small plasma volume/large surface area, where solid films are deposited. Other reactor systems (e.g., microwave systems) are also being developed.

A2-6.2 *Flow Capacity and Removal Capacity* — Reactor systems are designed for the process system effluent and are sized to handle specific process flows.

A2-6.3 *Efficiency* — With inputs of up to 300 sccm of silane, removal efficiencies of greater than 99% have been documented. At higher flows efficiency can be reduced. High removal efficiencies of PFCs have also been demonstrated.

A2-6.4 *Limitations* — Silane will form solids in the plasma. Gaseous by-products will pass into the exhaust. Solids transmission into the vacuum pump will be reduced, but additional abatement devices may be required downstream if by-products need to be removed. Formation of hydrogen ( $\text{H}_2$ ) as a by-product should be considered to ensure that the  $\text{H}_2$  outlet concentration is below the lower flammable limit.

### A2-7 Traps/Filters/Cyclones/Precipitators

A2-7.1 *Principle of Operation* — Designed to remove the particulate component in exhausts. Some processes produce solid powders (e.g., silica) and other processes generate condensable vapors. These vapors, on compression in the pump and cooling in the exhaust, condense into solid materials. Numerous designs exist to collect these solids out of the gas stream. These include filters (paper/bag etc.), cooled condensation chambers, cyclones, and electrostatic precipitators.

A2-7.2 *Removal Capacity* — Removal capacity is normally measured as the mass of solid the unit can collect before it partially blocks. As they are blocked, these systems generate a backpressure, which can prevent effective removal of material or create a process disturbance.

A2-7.3 *Efficiency* — Efficiency for these various units is difficult to measure. It is usually assessed pragmatically in terms of increased interval between exhaust clean out.

A2-7.4 *Limitations* — These various units must be used with care and only when not associated with hazardous gases. Often solid exhaust materials are mixed with hazardous gases. If these gases are present in the trapped solids a dangerous situation can result. Where gases and solids are mixed it is safer to keep material volatile and abate all by a POU abatement technology



from the sections above. If only non-hazardous gases are left these systems can provide cost-effective abatement to keep ducts clear. They can also be used in combination with other technologies.

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

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# SEMI F6-92

## GUIDE FOR SECONDARY CONTAINMENT OF HAZARDOUS GAS PIPING SYSTEMS

### 1 Purpose

To provide a guide for the design, fabrication, and operation of secondarily contained distribution piping for hazardous production material (HPM) gases.

### 2 Scope

This guide covers the general requirements for hazardous production material distribution piping in those industries that are included under the H-6 Classification of the Uniform Building Code, or Articles 51 or 80 of the Uniform Fire Code, or of other applicable local codes. This guide does not include requirements for individual exhausted enclosures (e.g., valve boxes and gas cabinets).

### 3 Applicable Documents

#### 3.1 SEMI Documents

SEMI F1 — Specification for Leak Integrity of Toxic Gas Piping Systems

SEMI F2 — Specification for Seamless Austenitic Stainless Steel Tubing for Semiconductor Manufacturing Applications

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

SEMI S2 — Safety Guidelines for Semiconductor Manufacturing Equipment

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

#### 3.2 ANSI/ASME Standard<sup>1</sup>

B31.3 — Chemical Plant and Petroleum Refinery Piping

#### 3.3 Federal Regulations<sup>2</sup>

29 CFR — Title 29 of the Code of Federal Regulations (CFR), Part 1910

49 CFR — Title 49 of the Code of Federal Regulations (CFR), Chapter I

#### 3.4 ICBO Codes<sup>3</sup>

UBC CH 9 — Chapter 9 of the Uniform Building Code, Requirements for Group H Occupancies

UFC ART 51 — Article 51 of the Uniform Fire Code, Semiconductor Fabrication Facilities Using Hazardous Production Materials

UFC STD 79-3 — Standard No. 79-3 of the Uniform Fire Code

UFC ART 80 — Article 80 of the Uniform Fire Code, Hazardous Materials

UFC ART 90 — Article 90 of the Uniform Fire Code, Regulation of Facilities Where Materials Which Are or May Become Toxic Gases Are Found

#### 3.5 NFPA Standards<sup>4</sup>

NFPA 70/ ART 500 — Article 500 of the National Electrical Code, Hazardous (Classified) Locations

NFPA 497M — Classification of Gases, Vapors and Dusts for Electrical Equipment in Hazardous (Classified) Locations

NFPA 704 — Identification of the Fire Hazards of Materials

#### 3.6 ACGIH Publication<sup>5</sup>

TLV<sup>6</sup> — American Conference of Governmental Industrial Hygienists Threshold Limit Values and Biological Exposure Indices

### 4 Terminology

4.1 *closed secondary containment* — Secondary containment that has a sealed annulus. In closed containment systems, the annular space either holds a certain pressure of gas or a certain level of vacuum. In closed containment, a change in the pressure or vacuum would be indicative of a leak in either the primary or secondary system.

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3 International Conference of Building Officials, 5360 South Workman Mill Road, Whittier, CA 90601

4 National Fire Protection Association, Batterymarch Park, Quincy, MA 02269

5 American Conference of Governmental Industrial Hygienists, 6500 Glenway Avenue, Building D-7, Cincinnati, OH 45211

6 TLV is a registered trademark of ACGIH.

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1 American National Standards Institute, 1430 Broadway, New York, NY 10018

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

4.2 *controlled work area* — A space within a building where HPM's may be stored, handled, dispensed, or used.

4.3 *equilibrium vapor concentration (EVC)* — The state of a material at which vapor pressure has stabilized and is no longer rising or falling. The EVC value (in parts per million) of a material is determined by multiplying the vapor pressure by  $10^6$  and dividing by atmospheric pressure (760 mmHg at sea level).

4.4 *hazardous production material (HPM)* — A solid, liquid, or gas that has a degree-of-hazard rating in health, flammability, or reactivity of Class 3 or 4 as ranked by NFPA 704 and that is used directly in research, laboratory, or production processes that have as their end product materials that are not hazardous.

4.5 *highly toxic gas* — A chemical that has a median lethal concentration ( $LC_{50}$ ) in air of 200 parts per million by volume or less of gas or vapor, or 2 milligrams per liter or less of mist, fume, or dust, when administered by continuous inhalation for one hour (or less if death occurs within one hour) to albino rats weighing between 200 and 300 grams each.

4.6 *immediately dangerous to life and health (IDLH)* — A concentration of airborne contaminants, normally expressed in parts per million or milligrams per cubic meter, which represents the maximum level from which one could escape within thirty minutes without any escape-impairing symptoms or irreversible health effects. This level is established by the National Institute of Occupational Safety and Health (NIOSH).

4.7 *level of concern (LOC)* — Equal to 0.1 of the IDLH value.

4.8 *lower detectable limit of instrument (LDL)* — The lowest concentration of a substance that will give an instrument response with a signal-to-noise ratio of at least 3 db.

4.9 *material hazard index (MHI)* — A numeric value used for ranking chemical production materials in order to determine the level of controls necessary for regulation. MHI is determined by dividing the equilibrium vapor concentration (EVC) of a material at 25°C by the level of concern (LOC) value for the material.

4.10 *open secondary containment* — Secondary containment with an open-ended annular space. This annulus must be directed to a system designed to handle the contained HPM.

4.11 *permissible exposure limit (PEL)* — The maximum permitted eight hour time-weighted average concentration of an airborne contaminant. The

maximum permitted time-weighted average exposures to be used are those published in 29 CFR 1910.1000.

4.12 *pressure decay method* — The method of detection of leakage through pressure loss, over a period of time, within a vessel or piping system.

4.13 *primary containment* — The first level of containment (i.e., the inside portion of the container that comes into immediate contact on its inner surface with the material being contained).

4.14 *pyrophoric* — Capable of spontaneous ignition in air at or below a temperature of 54.5°C (130°F).

4.15 *secondary containment* — Level of containment that is external to and separate from primary containment. Secondary containment is a method of safeguarding used to prevent unauthorized releases of toxic or hazardous gases into uncontrolled work areas. Secondary containment means those methods or facilities in addition to the primary containment system.

4.16 *threshold limit value/time-weighted average (TLV/TWA)* — As defined by the American Conference of Governmental Industrial Hygienists (ACGIH).

4.17 *uncontrolled work area* — Any area outside of a secondary containment system where people are likely to be present.

4.18 *vacuum decay method* — Leakage detection determined by the loss of vacuum (increase in pressure), over a period of time within a vessel or piping system.

## 5 Containment Function

5.1 Secondary containment should, in case of release, segregate the hazardous production material (HPM) from the surrounding area.

5.2 Secondary containment systems should include provisions to contain and detect substances which pose health or property hazards and to direct such substances into areas or facilities that can safely treat, dispose of, or dilute them prior to release into the atmosphere.

## 6 Containment Application

Secondary containment may be used on any system if deemed appropriate by the authority responsible for the system. Secondary containment should be mandatory for all HPM's that are included in the following categories:

6.1 Those substances with threshold limit values (TLV) below the lower detectable limit (LDL) of the detection systems in use.

6.2 Those substances that provide inadequate warning properties.

6.3 Those substances that fall into the following categories:

6.3.1 Any HPM's which have a material hazard index (MHI) value equal to or greater than 500,000.

6.3.2 Highly toxic gas.

6.3.3 Pyrophorics.

6.3.4 Substances with unknown, but potentially high toxicities (e.g., organometallics).

6.3.5 Materials with an NFPA 704 reactivity rating of 3 or 4.

6.3.6 Any corrosives that are not contained in inert process piping.

6.4 Where piping containing HPM's is installed in a manner to conceal it from view.

6.5 Where piping containing HPM's is installed in uncontrolled or unventilated areas such as drop ceilings or behind walls.

6.6 Where required by UBC H-6 or other existing codes.

6.7 Where there is any reasonable possibility of a leak in the primary containment due to normal wear and tear, possible abuse, or corrosive attack to the piping on the inside or exterior.

## 7 Containment Methods

The method of containment may take many forms provided that the method is sound in engineering design. This design should be in conformance to nationally recognized codes and standards as well as to the requirements of local safety jurisdictions.

## 8 Materials of Construction

8.1 *Chemical Compatibility* — If there is a possibility that the HPM, or its reaction products, is corrosive, to any extent, to the secondary containment, one should develop a procedure to verify the integrity of the containment system. This verification procedure need only be performed in the event that the HPM, or its reaction products, contacts the containment (see Section 9).

8.2 *Fire Resistance* — If walls are used as the secondary containment system, they should be constructed of materials resistant of fire. The fire rating of the secondary containment should be as follows:

8.2.1 Two hour rating for pyrophorics;

8.2.2 One hour rating for all others.

## 9 Design Requirements

9.1 *Design Pressure* — The system should be designed to provide secondary containment with the ability to withstand the pressure of the entire volume of the potential leaking source without leakage.

9.1.1 Closed secondary containment systems should have sufficient design pressure to withstand the pressure of the entire contents of the source of the HPM. The design safety factor must be consistent with ANSI B31.3.

9.1.2 Open secondary containment systems should have sufficient design pressure to withstand the release at full tank pressure or the entire contents of the HPM released within a time of two minutes.

9.1.3 Special consideration should be given to the containment of pyrophorics. Some pyrophorics can develop considerable explosive pressure. The elimination of oxygen from the annulus of a containment system should be considered in lieu of explosive pressure data.

9.2 *Resistance to External Forces* — The method of construction of secondary containment systems should be sufficient to withstand:

9.2.1 Normal physical abuse found in the industrial workplace.

9.2.2 Seismic zone activity as shown in the UBC (Uniform Building Code) or other applicable codes.

9.3 *Leak Integrity* — The system should be designed to provide secondary containment with sufficient leak integrity to prevent exceeding the PEL of the HPM in uncontrolled work areas.

9.3.1 Primary piping should be leak tested in accordance with SEMI F1.

9.3.2 Closed secondary containment systems should have the same leak integrity as the primary containment except the closed secondary containment systems should be leak tested with the annulus pressurized to 2 times the maximum operating pressure with argon containing at least 10% helium. There should be no drop in pressure for a period of 12 hours.

9.3.3 Open secondary containment systems should be leak tested to the same criteria as closed secondary containment systems per 9.3.2 above.

## 10 Separation of HPM's

A single secondary containment may be used to contain more than one HPM provided that those HPM's are not reactive with any other HPM in the common containment. Documents that should be considered for proper separation of HPM's include:

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

10.2 Materials Safety Data Sheets

10.3 NFPA Fire Protection Guide on Hazardous Materials

## 11 Cross Connections

There should be no mechanical cross connection of secondary containment systems with other systems not designed specifically for the secondary containment of the particular HPM's.

## 12 Monitoring

The annulus should be monitored for leakage of the primary system in accordance with the following:

12.1 The sensitivity of the detection system should be sufficient to detect leakage at 1/2 Threshold Limit Values (TLV's) at the discharge to treatment of an open secondary containment system.

12.2 Detection of leakage into the annulus of the secondary containment system should include alarm systems.

12.3 Detection methods may include:

12.3.1 Direct detection of the HPM or its reaction products (open secondary containment system).

12.3.2 Pressure decay method (closed secondary containment system).

12.3.3 Vacuum decay method (closed secondary containment system).

## 13 Leak Management

The secondary containment system should be designed to control and direct leaking materials. Control of HPM's may consist of dilution, absorption, incineration, scrubbing, venting, or those methods deemed safe and suitable to the governing authorities responsible for the facility. The initiation of the secondary containment alarm system should be automated. This system should automatically institute the management of the leaking HPM in the event of a breach of the primary system.

## 14 Periodic Testing - After Installation

14.1 Secondarily contained piping systems must be inspectable. The method of inspection must be able to reveal the current strength and leak integrity of the primary and secondary containment systems.

14.2 The secondary containment must be periodically leak tested in accordance with the criteria set forth in Section 9.3 of the above. Structural testing of both the process piping and the secondary containment should be conducted to the maximum pressure specified in Section 9.1 of the above.

14.3 Any secondarily contained piping system failing the periodic inspection should be repaired or replaced immediately.

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

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

# **SEMI F7-92 (Reapproved 0299)**

## **TEST METHOD TO DETERMINE THE TENSILE STRENGTH OF TUBE FITTING CONNECTIONS MADE OF FLUOROCARBON MATERIALS**

This test method was technically reapproved by the Facilities Committee and is the direct responsibility of the North American Facilities Committee. Current edition approved by the North American Regional Standards Committee in October 1998. Initially available on [www.semi.org](http://www.semi.org) February 1999; to be published February 1999. Originally published in 1992.

### **1. Purpose**

1.1 This method provides a uniform procedure to determine the tensile strength of tube fitting connections made of fluorocarbon materials.

### **2. Scope**

2.1 This method can be used to characterize tube fitting connections on the basis of test data developed under the conditions described herein, but the results are not intended to imply a performance rating.

2.2 Tube defined in this method has a circular cross section and is made of fluorocarbon materials.

2.3 All parts of the tube fittings tested by this method in contact with the internal fluid are made of fluorocarbon materials.

2.3.1 Parts such as a nuts or grippers are not limited to being made of a fluorocarbon material.

2.4 Only the seal between the tube and tube fitting being evaluated is within the scope of this document. All other, threaded seals are beyond the scope of this document.

2.5 When using this method for making comparisons among various tube fittings and/or manufacturers, the user must be specific in the selection of the tube and tube fittings to be evaluated.

2.6 The International System of Units (SI) is used as the standard unit of measure in this document. The U.S. Customary units are in parentheses for reference purposes only and have been rounded to the nearest whole value.

### **3. Referenced Documents**

#### **3.1 ASTM Standards<sup>1</sup>**

*D3307 PFA* — Fluorocarbon Molding and Extrusion Materials

*D3296* — Standard Specification for FEP Fluorocarbon Tube

### **4. Summary of Method**

4.1 Subject tube fitting connections made of fluorocarbon materials to extreme tensile forces.

### **5. Significance and Use**

5.1 In the manufacturing of semiconductor products, many types of hazardous chemicals and solvents are required. As a result, tubing and various fitting designs of fluorocarbon materials (which are chemically resistant to these fluids) are used to transport those fluids. It is important to control the testing process when evaluating various fitting designs, so that accurate comparisons of the tensile strength can be made.

5.2 It is the intent of this method to provide a procedure in which the tensile force will be applied to tube fitting connections made of fluorocarbon materials. By using this method, accurate comparisons of various tube fitting designs can be achieved.

5.3 The results obtained when using this method are applicable only to conditions that specifically duplicate the procedures used within this method.

5.4 When using this test method, it is assumed that the test specimens are truly representative of the material and manufacturing process specified for that product. Departure from this assumption could introduce discrepancies that are greater than those introduced by departure from the details of the procedure outlined in this method.

### **6. Terminology**

6.1 *characterize* — To describe the quality of.

6.2 *failure* — Tube separation from a tube fitting connection or tearing of the tube.

6.3 *subject* — To expose to.

6.4 *tensile* — Longitudinal, so as to lengthen the test object.

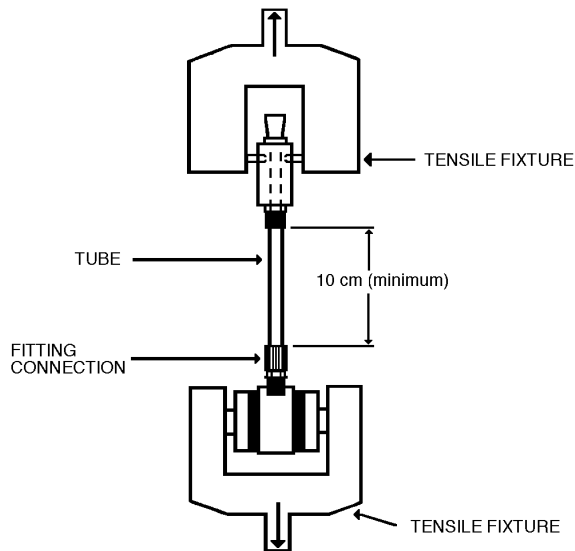
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<sup>1</sup> American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959

## 7. Description of Test Equipment

7.1 A test apparatus capable of securing the test specimen while accurately providing a uniform rate of pull to the specimen. The test instrument shall have the capability of recording the maximum tensile force applied to the test specimen.

7.2 See Figure 1 for basic tensile test apparatus.



**Figure 1**  
**Basic Tensile Test Apparatus**

## 8. Safety Precautions

**WARNING:** This test method will subject test specimens to conditions that may exceed the normal performance rating of the products under evaluation. Adequate precautions must be taken to prevent injury to the person conducting the test.

## 9. Test Specimens and Conditioning

9.1 *Sample Size* — A minimum of three specimens shall be tested.

9.2 *Specimen Size* — The specimen length shall be no less than 10 cm (4 in) between the tensile fixture and the tube end of the fitting connection.

9.3 *Specimen Surface* — All surfaces of the specimens shall be free of visible flaws, scratches, or other imperfections, unless typically found on a representative sample of the product.

9.4 *Specimen Conditioning* — All specimens must be conditioned for a minimum of one hour in an air

environment of  $23^{\circ} \pm 2.8^{\circ}\text{C}$  ( $73^{\circ} \pm 5^{\circ}\text{F}$ ) prior to being subjected to tensile forces.

## 10. Calibration

10.1 Calibrate the tensile test equipment's rate of pull to 2.5 cm (1 in) per minute,  $\pm 5\%$ .

10.2 Calibrate the tensile force instrument to  $\pm 2\%$  full scale.

## 11. Test Procedure

11.1 Assemble a test fitting connection, per manufacturer's specification, to one end of the tube. (See 9.2.)

11.2 Install the fitting connection to the tensile fixture (see Figure 1) in a manner that prevents distortion of the connection.

11.3 Secure the open end of the tube to the tensile fixture, leaving a minimum of 10 cm (4 in) of exposed tube.

11.4 Begin pulling the test specimen at a rate of 2.5 cm (1 in) per minute.

11.5 Continue applying tensile force until the tube pulls through the fitting connection or until tearing of the tube occurs.

11.5.1 If the tube releases from the tensile fixture, the data from that specimen must be disregarded and an additional specimen tested.

11.6 Record on test data sheet (see Appendix 1 for a sample) the mode of failure and the maximum tensile force applied, in Newtons (lbs).

## 12. Calculations

12.1 Calculate the average "maximum tensile force" and record.

12.2 Calculate the standard deviation of "maximum tensile force" and record.

## 13. Data Accuracy

13.1 *Tensile Force* — Newtons:  $\pm 2\%$ .

## 14. Test Data Sheet

The test data sheet shall include the following information:



14.1 Date tested.

14.2 Operator and test facility.

14.3 Description of items tested, including:

*Tubing* — manufacturer, O.D., wall thickness, part number, and material type.

*Fitting* — manufacturer, type, size, part number, and material type.

14.4 Maximum tensile force for each specimen.

14.5 Average and standard deviation of maximum tensile forces.

14.6 Mode of failure for each specimen.

NOTE: A sample test data sheet is provided as Appendix 1.

## APPENDIX 1

### SAMPLE TEST DATA SHEET

NOTE: This related information is not an official part of this SEMI test method and is not intended to modify or supercede the official test method. It has been derived from industry specifications. Publication was authorized by full letter ballot procedures. Determination of the suitability of the material is solely the responsibility of the user.

Test Date:	Operator:	Test Facility:
Tube Manufacturer:		Fitting Manufacturer:
Tube O.D.:		Fitting Type:
Tube Wall Thickness:		Fitting Size:
Tube P/N:		Fitting P/N:
Tube Material:		Fitting Material:

Tensile Force — Newtons

<i>Sample</i>	<i>Maximum Tensile Force</i>	<i>Mode of Failure</i>
1		
2		
3		
Average =		
Standard Deviation =		

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

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# **SEMI F8-0998**

## **TEST METHOD FOR EVALUATING THE SEALING CAPABILITIES OF TUBE FITTING CONNECTIONS MADE OF FLUOROCARBON MATERIALS, WHEN SUBJECTED TO TENSILE FORCES**

### **1 Purpose**

1.1 This method provides a uniform procedure to determine the sealing capabilities of tube fitting connections made of fluorocarbon materials when the connections are subjected to tensile forces.

### **2 Scope**

2.1 This method can be used to characterize tube fitting connections on the basis of test data developed under the conditions described herein, but the results are not intended to imply a performance rating.

2.2 Tube defined in this method has a circular cross section and is made of fluorocarbon materials.

2.3 All parts of the tube fittings tested by this method in contact with the internal fluid are made of fluorocarbon materials.

2.3.1 Parts such as a nut or gripper are not limited to being made of a fluorocarbon material.

2.4 Only the seal between the tube and tube fitting being evaluated is within the scope of this document. All other, threaded seals are beyond the scope of this document.

2.5 When using this method for making comparisons between various tube fittings and/or manufacturers, the user must be specific in the selection of the tube and tube fittings to be evaluated.

2.6 The International System of Units (SI) is used as the standard unit of measure in this document. The U.S. Customary units are in parentheses for reference purposes only and have been rounded to the nearest whole value.

### **3 Referenced Documents**

#### **3.1 *ASTM Standards*<sup>1</sup>**

D 3307 PFA — Fluorocarbon Molding and Extrusion Materials

D 3296 — Standard Specification for FEP Fluorocarbon Tube

### **4 Summary of Method**

4.1 Subject tube fitting connections made of fluorocarbon materials to extreme tensile forces, while maintaining a specified pressure to the internal cavity of the fitting and tube.

### **5 Significance and Use**

5.1 In the manufacturing of semiconductor products, many types of hazardous chemicals and solvents are required. As a result, tubing and various fitting designs made of fluorocarbon materials (which are chemically resistant to these fluids) are used to transport those fluids. It is important to control the testing process when evaluating various fitting designs, so that accurate comparisons of the sealing capabilities of tube fitting connections can be made.

5.2 It is the intent of this method to provide a procedure in which the air pressure and tensile force will be applied to tube fitting connections made of fluorocarbon materials. By using this method, accurate comparisons of various tube fitting designs can be achieved.

5.3 The results obtained when using this method are applicable only to conditions that specifically duplicate the procedures used within this method.

5.4 When using this test method, it is assumed that the test specimens are truly representative of the material and manufacturing process specified for that product. Departure from this assumption could introduce discrepancies that are greater than those introduced by departure from the details of the procedure outlined in this method.

### **6 Terminology**

#### **6.1 *Acronyms***

6.1.1 *O.D.* — Outside diameter

6.1.2 *P/N* — Part number

#### **6.2 *Definitions***

6.2.1 *characterize* — To describe the quality of.

6.2.2 *failure* — Tube separation from a tube fitting connection or tearing of the tube.

6.2.3 *standard deviation* — A measure of the variation among the members of a statistical sample.

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<sup>1</sup> American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959

6.2.4 *submersion tank* — A transparent tank filled with isopropyl alcohol to allow observation of air leakage from the tube fitting connection.

6.2.5 *subject* — To expose to.

6.2.6 *tensile* — Longitudinal, so as to lengthen the test object.

## 7 Description of Test Equipment

7.1 A supply of compressed air. Pressure to be within  $\pm 2\%$  of specified.

7.2 Isopropyl alcohol.

7.3 An instrument to record the tensile force applied to the test specimen.

7.4 A test apparatus:

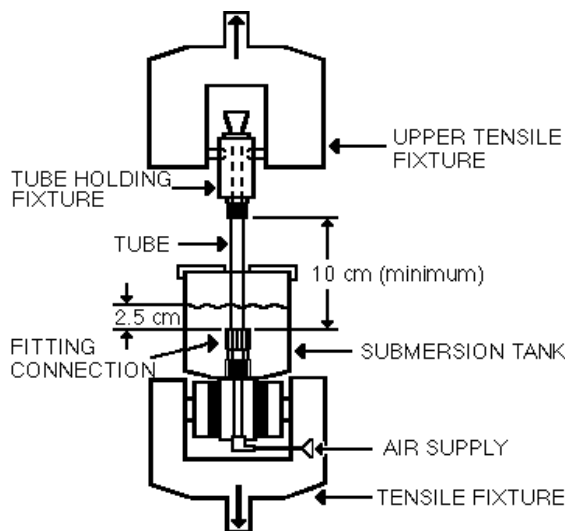
7.4.1 Capable of securing the test specimen while allowing the specimen to be internally pressurized.

7.4.2 Capable of accurately providing a uniform rate of pull to the specimen.

7.4.3 With a transparent submersion tank capable of containing a fluid for observing leakage. The minimum fluid level shall be 2.5 cm (1 in) above the test connection.

7.4.4 The apparatus shall allow the test specimen to be internally pressurized to 250 kPa (36 psig).

7.5 See Figure 1 for basic tensile and leak test apparatus.



**Figure 1**  
**Basic Tensile and Leak Test Apparatus**

## 8 Safety Precautions

**WARNING: This test method will subject test specimens to conditions that may exceed the normal performance rating of the products under evaluation. Adequate precautions must be taken to prevent injury to the person conducting the test.**

## 9 Test Specimens and Conditioning

9.1 *Sample Size* — A minimum of three specimens shall be tested.

9.2 *Specimen Size* — The specimen length shall be no less than 10 cm (4 in) between the upper tensile fixture and the tube end of the fitting connection.

9.3 *Specimen Surface* — All surfaces of the specimens shall be free of visible flaws, scratches, or other imperfections, unless typically found on a representative sample of the product.

9.4 *Specimen Conditioning* — All specimens must be conditioned for a minimum of one hour in an air environment of  $23^{\circ} \pm 2.8^{\circ}\text{C}$  ( $73^{\circ} \pm 5^{\circ}\text{F}$ ) prior to being subjected to pressure and tensile forces.

## 10 Calibration

10.1 Calibrate the tensile test equipment's rate of pull to 2.5 cm (1 in) per minute,  $\pm 5\%$ .

10.2 Calibrate the tensile force instrument to  $\pm 2\%$  full scale.

## 11 Test Procedure

11.1 Assemble a test fitting connection, per manufacturer's specification, to one end of the tube.

11.2 Install the fitting connection to the tensile fixture, in the submersion tank (see Figure 1) in a manner that prevents distortion of the connection.

11.3 Secure free end of tube to the upper tensile fixture, leaving a minimum of 10 cm (4 in) of exposed tube.

11.4 Fill the submersion tank with isopropyl alcohol to a height of 2.5 cm (1 in) above the top of the test connection.

11.5 Cover the submersion tank in case of a fluid splash.

11.5.1 The cover must allow for slight clearance around tube.

11.6 Pressurize the test specimen with an internal air pressure of 250 kPa (36 psig), regardless of tube size.

11.7 Begin pulling the test specimen at a rate of 2.5 cm (1 in) per minute.

11.8 Record on test data sheet (see Related Information 1 for a sample), the minimum tensile force in Newtons (lbs) when leakage is first observed from the fitting connection.

11.9 Lower the air pressure to one atmosphere immediately after observing any leakage.

11.10 Continue pulling until the tube pulls through the fitting connection or until tearing of the tube occurs.

11.10.1 If the tube releases from the tensile fixture, the data from that specimen must be disregarded and an additional specimen tested.

11.11 Record on test data sheet the mode of failure and the maximum tensile force applied in Newtons (lbs).

11.11.1 It is possible that the tensile force required to cause leakage and the maximum tensile force may be the same. If this situation occurs, indicate the force reading on both areas of the test data sheet.

## 12 Calculations

12.1 Calculate the average “force to cause leakage” and record.

12.2 Calculate the average “maximum tensile force” and record.

12.3 Using the three actual forces:

12.3.1 Calculate the standard deviation of “force to cause leakage” and record.

12.3.2 Calculate the standard deviation of “maximum tensile force” and record.

## 13 Data Accuracy

13.1 *Tensile Force* — Newtons:  $\pm 2\%$

## 14 Test Data Sheet

The test data sheet shall include the following information:

14.1 Date tested.

14.2 Operator and test facility.

14.3 Description of items tested, including:

*Tubing* — manufacturer, O.D., wall thickness, part number, and material type.

*Fitting* — manufacturer, type, size, part number, and material type.

14.4 Force to cause leakage for each specimen.

14.5 Average and standard deviation of the forces to cause leakage.

14.6 Maximum tensile force for each specimen.

14.7 Average and standard deviation of maximum tensile forces.

14.8 Mode of failure for each specimen.

NOTE: A sample test data sheet is provided as Related Information 1.

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

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## RELATED INFORMATION 1 SAMPLE TEST DATA SHEET

NOTE: This related information is not an official part of SEMI F8 and is not intended to modify or supercede the official test method. It has been derived from industry specifications. Publication was authorized by full letter ballot procedure. Determination of the suitability of the material is solely the responsibility of the user.

Test Date:	Operator:	Test Facility:
Tube Manufacturer:		Fitting Manufacturer:
Tube O.D.:		Fitting Type:
Tube Wall Thickness:		Fitting Size:
Tube P/N:		Fitting P/N:
Tube Material:		Fitting Material:

Tensile Force — Newtons

<i>Specimen</i>	<i>Force to Cause Leakage</i>	<i>Maximum Tensile Force</i>	<i>Mode of Failure</i>
1			
2			
3			
Average =			
Standard Deviation =			

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# **SEMI F9-0998**

## **TEST METHOD TO DETERMINE THE LEAKAGE CHARACTERISTICS OF TUBE FITTING CONNECTIONS MADE OF FLUOROCARBON MATERIALS, WHEN SUBJECTED TO A SIDE LOAD CONDITION**

### **1 Purpose**

1.1 This method provides a uniform procedure to determine the leakage characteristics of tube fitting connections made of fluorocarbon materials, when subjected to side loading.

### **2 Scope**

2.1 This method can be used to characterize tube fitting connections on the basis of test data developed under the conditions described herein, but the results are not intended to imply a performance rating.

2.2 Tube defined in this method has a circular cross section and is made of fluorocarbon materials.

2.3 All parts of the tube fittings tested by this method in contact with the internal fluid are made of fluorocarbon materials.

2.3.1 Parts such as a nut or gripper are not limited to being made of a fluorocarbon material.

2.4 Only the seal between the tube and tube fitting being evaluated is within the scope of this document. All other, threaded seals are beyond the scope of this document.

2.5 When using this method for making comparisons between various tube fittings and/or manufacturers, the user must be specific in the selection of the tube and tube fittings to be evaluated.

2.6 The International System of Units (SI) is used as the standard unit of measure in this document. The U.S. Customary units are in parentheses for reference purposes only and may have been rounded to the nearest whole value.

### **3 Summary of Method**

3.1 Subject tube fitting connections made of fluorocarbon materials to a side load condition that is a result of bending the tube in a uniform arc, while maintaining a specified pressure to the internal cavity of the fitting and tube.

### **4 Significance and Use**

4.1 In the manufacturing of semiconductor products, many types of hazardous chemicals and solvents are required. As a result, tubing and various fitting designs made of fluorocarbon materials (which are chemically

resistant to these fluids) are used to transport those fluids. It is important to control the testing process when evaluating various fitting designs, so that accurate comparisons of the side load capabilities can be made.

4.2 It is the intent of this method to provide a procedure in which tube fitting connections made of fluorocarbon materials will be subjected to extreme side loading. By using this method, accurate comparisons of various tube fitting designs can be achieved.

4.3 The results obtained when using this method are applicable only to conditions that specifically duplicate the procedures used within this document.

4.4 When using this test method, it is assumed that the test specimens are truly representative of the material and manufacturing process specified for that product. Departure from this assumption could introduce discrepancies that are greater than those introduced by departure from the details of the procedure outlined in this method.

### **5 Terminology**

#### *5.1 Acronyms*

5.1.1 *O.D.* — Outside diameter

5.1.2 *P/N* — Part number

#### *5.2 Definitions*

5.2.1 *bend radius* — The distance from the center of an imaginary circle on which the arc of the bent tube falls to a point on the arc.

5.2.2 *characterize* — To describe the quality of.

5.2.3 *kinking* — A collapse of the tube wall caused by excessive bending.

5.2.4 *“L”* — The tube length required to produce a uniform 180° arc, at a specified tube bend radius.

NOTE: The symbol “L” is unique in that it is used only in this test method.

5.2.5 *“R”* — The theoretical value to determine the tube bend radius, used during testing.

NOTE: The symbol “R” is unique in that it is used only in this test method.

5.2.6 *side load* — A result of bending a tube in a specified arc, consequently subjecting the tube fitting connection to a radial stress.

5.2.7 *subject* — To expose to.

5.2.8 *submersion tank* — A transparent tank, filled with isopropyl alcohol, to allow observation of air leakage from a tube fitting connection.

## 6 Description of Test Equipment

6.1 A supply of compressed air. Pressure to be within  $\pm 2\%$  of specified value.

6.2 Isopropyl alcohol.

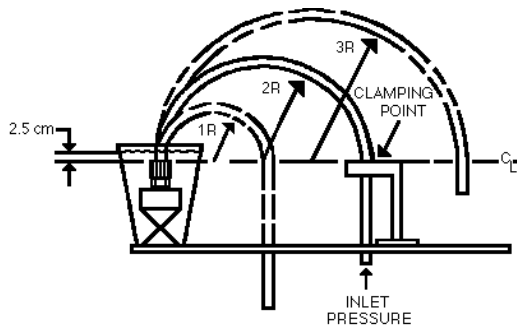
6.3 An instrument for measuring lengths of tubing (e.g., tape measure).

6.4 A special apparatus which has a fixture for attaching the fitting body, while also providing a means to clamp the free end of tube, to maintain a tube bend radius.

6.4.1 The apparatus shall allow the test specimen to be internally pressurized.

6.4.2 The apparatus shall have a transparent submersion tank capable of containing a fluid for observing leakage. The minimum fluid level shall be 2.5 cm (1 in) above the test connection.

6.5 See Figure 1 for a basic side load test apparatus.



**Figure 1**  
**Side Load Test Apparatus**

## 7 Safety Precautions

**WARNING:** This test method will subject test specimens to conditions that may exceed the normal performance rating of the products under evaluation. Adequate precautions must be taken to prevent injury to the person conducting the test.

## 8 Test Specimens and Conditioning

8.1 *Sample Size* — A minimum of three specimens shall be tested.

8.2 *Tube* — The tubing used for this evaluation shall be of straight lengths, not previously coiled.

8.3 *Specimen Surface* — All surfaces of the specimens shall be free of visible flaws, scratches, or other imperfections, unless typically found on a representative sample of the product.

8.4 *Specimen Conditioning* — All specimens must be conditioned for a minimum of one hour in an air environment of  $23^{\circ} \pm 2.8^{\circ}\text{C}$  ( $73^{\circ} \pm 5^{\circ}\text{F}$ ) prior to being subjected to pressurization and bending of the tube.

## 9 Test Procedure

9.1 Perform the following calculation to determine the “R” value.

9.1.1 For a tubing outside diameter (O.D.) of 1.5 cm (.59 in) or less:  $R = \text{Tube O.D.} \times 5$ .

9.1.2 For a tubing outside diameter (O.D.) greater than 1.5 cm (0.59 in):  $R = \text{Tube O.D.} \times 5$ .

NOTE: The constants (5 and 10) in Sections 9.1.1 and 9.1.2 were derived from the extrapolation of the actual “Minimum Bend Radius” of tubing from various manufacturers.

9.2 Perform the calculation in Section 9.2.1 to determine the tube length required to produce a  $180^{\circ}$  arc at a tube bend radius of R.

9.2.1  $L = R \times 3.14$

9.3 Cut three tubes of equal length per the following formula:  $3L + 15 \text{ cm (6 in)}$ .

9.4 Install a fitting body to the test fixture, in the submersion tank.

9.5 Install a tube into the fitting body and assemble the connection per manufacturer’s specification.

NOTE: To prevent side loading of the fitting connection prior to bending of the tube, provide a method of supporting the tube.

9.6 Measure from the tube end of the fitting connection and place a mark on the tube (within  $\pm 1\%$  of calculated value) in five separate locations as determined below:

- 0.5L
- 1.0L
- 1.5L
- 2.0L
- 3.0L

9.7 Use the following formula to determine the actual tube bend radius used and record on test data sheet (see Related Information 1 for a sample):



- 0.5R
- 1.0R
- 1.5R
- 2.0R
- 3.0R

9.8 Fill the submersion tank with isopropyl alcohol to a minimum height 2.5 cm (1 in) above the top of the test connection.

9.9 Pressurize the test specimen with an internal air pressure of 250 kPa (36 psig), regardless of tube size.

9.10 Observe the connection for a minimum of one minute for bubble leakage (bbl/min) and record on test data sheet under the column entitled "STRAIGHT".

9.11 Carefully bend the tube in a uniform 180° arc with a radius of 3R and clamp the free end to the test fixture. (See Section 9.7 for specific R value.) Proper tube length at the clamping point is determined in Section 9.6.

9.11.1 R value tolerances =

- Tubes with 1.5 cm (0.59 in) O.D. or less =  $\pm 5\%$
- Tubes with greater than 1.5 cm (0.59 in) O.D. =  $\pm 1\%$

9.12 Observe the connection for a minimum of one minute for bubble leakage. Record leakage on the appropriate area of the test data sheet. If no leakage is observed, note as "None".

9.13 Repeat Sections 9.11 and 9.12 for the remaining tube bend radius of 2R, 1.5R, 1R, and 0.5R, in descending order.

9.13.1 Should any tube kink prior to achieving 0.5R, testing of that sample will be discontinued. The radius at which kinking occurred shall be recorded and noted as "kink".

9.13.2 To reduce the effects caused by creep of the material the total time from initial bending of the tube until achieving the 0.5R value is to be less than ten minutes.

9.14 After completion of the first sample, replace the fitting and tube with new specimens and repeat Sections 9.4 through 9.13.2 for a total of three samples.

## 10 Test Data Sheet

The test data sheet shall include the following information:

10.1 Date tested.

10.2 Operator and test facility.

10.3 Description of items tested, including:

*Tubing* — Manufacturer, O.D., wall thickness, part number, and material type.

*Fitting* — Manufacturer, type, size, part number, and material type.

10.4 The actual tube bend radius for each nominal bend radius.

10.5 The leakage rate, in bubbles per minute, for each sample at each radius.

NOTE: A sample test data sheet is provided as Related Information 1.

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## RELATED INFORMATION 1 SAMPLE TEST DATA SHEET

NOTE: This related information is not an official part of SEMI F9 and is not intended to modify or supercede the official test method. It has been derived from industry specifications. Publication was authorized by full letter ballot procedures. Determination of the suitability of the material is solely the responsibility of the user.

Test Date:	Operator:	Test Facility:
Tube Manufacturer:		Fitting Manufacturer:
Tube O.D.:		Fitting Type:
Tube Wall Thickness:		Fitting Size:
Tube P/N:		Fitting P/N:
Tube Material:		Fitting Material:

Leakage— (bbl/min.)      [    ] = Actual Tube Bend Radius, cm(s)

		<i>3R</i>	<i>2R</i>	<i>1.5R</i>	<i>1R</i>	<i>0.5R</i>
Sample	Straight	[    ]	[    ]	[    ]	[    ]	[    ]
1						
2						
3						

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# SEMI F10-0698

## TEST METHOD TO DETERMINE THE INTERNAL PRESSURE REQUIRED TO PRODUCE A FAILURE OF A TUBE FITTING CONNECTION MADE OF FLUOROCARBON MATERIALS

### 1 Purpose

1.1 This method provides a uniform procedure to determine the internal pressure required to produce failure of fitting connections made of fluorocarbon materials.

### 2 Scope

2.1 This method can be used to characterize tube fitting connections on the basis of test data developed under the conditions described herein, but the results are not intended to imply a performance rating.

2.2 Tube defined in this method has a circular cross section and is made of fluorocarbon materials.

2.3 Tube fittings defined in this method are made of fluorocarbon materials for all parts in contact with the internal fluid.

2.3.1 Parts such as a nut or gripper are not limited to being made of a fluorocarbon material.

2.4 Only the seal between the tube and tube fitting being evaluated is within the scope of this document. All other threaded seals are beyond the scope of this document.

2.5 When using this method for making comparisons among various tube fittings and/or manufacturers, the user must be specific in the selection of the tube and tube fittings to be evaluated.

2.6 The International System of Units (SI) is used as the standard unit of measure in this document. The U.S. Customary units are in parentheses for reference purposes only and may have been rounded to the nearest whole value.

### 3 Referenced Document

#### 3.1 *ASTM Standard*<sup>1</sup>

ASTM D 1599 — Test Method for Short-Time Hydraulic Failure Pressure of Plastic Pipe, Tubing, and Fittings

### 4 Summary of Method

4.1 This test method consists of pressurizing tube fitting connections made of fluorocarbon materials to

failure by continuously increasing the internal hydraulic pressure. New tubing and fitting samples are required at each specific elevated fluid temperature condition, and failures are to occur within 60 to 70 seconds from initial exposure to the high pressure fluid.

### 5 Significance and Use

5.1 In the manufacturing of semiconductor products, many types of hazardous chemicals and solvents are required. As a result, tubing and various fitting designs made of fluorocarbon materials (which are chemically resistant to these fluids) are used to transport those fluids. It is important to control the testing process when evaluating various fitting designs, so that accurate comparisons of the pressure limits can be made.

5.2 It is the intent of this method to provide a procedure to determine the maximum pressure limits of tube fitting connections made of fluorocarbon materials. By using this method, accurate comparisons of various tube fitting designs can be achieved.

5.3 The results obtained when using this method are applicable only to conditions that specifically duplicate the procedures used within this method.

5.4 When using this test method, it is assumed that the test specimens are truly representative of the material and manufacturing process specified for that product. Departure from this assumption could introduce discrepancies that are greater than those introduced by departure from the details of the procedure outlined in this method.

### 6 Terminology

6.1 *characterize* — To describe the quality of.

6.2 *failure* — Any external leakage of fluid through the tube wall or the tube fitting connection, whether it be catastrophic or a slow leak.

6.3 *free end closure* — A metal tube fitting connection which is securely fastened to the tube and does not contribute to the restraint of the test specimen.

6.4 *isolation valve* — A valve used to separate the high temperature fluid from the high pressure fluid or to separate the samples from each other.

NOTE: This definition is used in only this test method.

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<sup>1</sup> American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959

6.5 *pressure containing envelope* — The internal area of a specimen which contains the fluid media.

6.6 *standard deviation* — A measure of the variation among the members of a statistical sample.

6.7 *subject* — To expose to.

## 7 Description of Test Equipment

7.1 This test method requires the use of two hydraulic systems.

7.1.1 A high pressure system capable of applying a continuous increase of the internal hydraulic pressure to the test specimen.

7.1.2 A low pressure, high temperature circulation system capable of controlling a fluid temperature within  $\pm 3^{\circ}\text{C}$  ( $\pm 5^{\circ}\text{F}$ ) of a specified test temperature.

7.1.2.1 The pressure is to be 75 kPa (11 psig) or less for all temperatures, within the range of  $25^{\circ}$  to  $200^{\circ}\text{C}$  ( $77^{\circ}$  to  $392^{\circ}\text{F}$ ).

7.2 *Test Fluid* — When testing at or below  $82^{\circ}\text{C}$  ( $180^{\circ}\text{F}$ ), an SAE 20 weight hydraulic oil may be used. When testing at elevated fluid temperatures, use a synthetic oil that is acceptable for elevated temperatures of up to  $200^{\circ}\text{C}$  ( $392^{\circ}\text{F}$ ) and compatible with the test stand and test specimens.

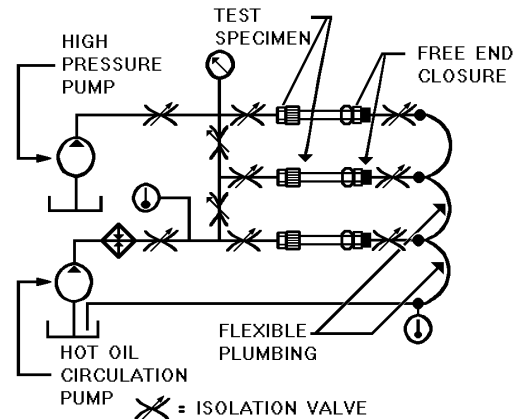
7.3 An instrument to record the peak pressure within 1% of full scale.

7.4 An instrument to monitor temperature within  $\pm 1^{\circ}\text{C}$  ( $\pm 2^{\circ}\text{F}$ ).

7.5 *Specimen Support* — Any support is acceptable as long as it does not contribute to the restraint of the specimen in either the circumferential or axial direction.

7.6 An enclosure that will allow observation of the test specimen and that is also capable of controlling the ambient temperature to  $31^{\circ} \pm 6^{\circ}\text{C}$  ( $88^{\circ} \pm 11^{\circ}\text{F}$ ).

7.7 See Figure 1 for basic pressure and circulation system.



**Figure 1**  
**Basic Pressure and Circulation System**

## 8 Safety Precautions

**WARNING:** This test method will subject test specimens to conditions that exceed the normal performance rating of the product under evaluation. Adequate precautions must be taken to prevent injury to the person conducting the test, as catastrophic failures of the test specimens are expected. The hydraulic plumbing and safety enclosure is to be constructed of materials which are capable of containing a fluid at  $200^{\circ}\text{C}$  ( $392^{\circ}\text{F}$ ).

## 9 Test Specimens and Conditioning

9.1 *Sample Size* — A minimum of three specimens shall be tested at each temperature condition.

9.1.1 The three connections will be conditioned simultaneously. See Figure 1 for a basic pressure and circulation system.

9.2 *Specimen Surface* — All surfaces of the specimens shall be free of visible flaws, scratches, or other imperfections, unless typically found on a representative sample of the product.

9.3 *Specimen Conditioning* — All specimens must be conditioned for a minimum of one hour in an air environment of  $23^{\circ} \pm 2.8^{\circ}\text{C}$  ( $73^{\circ} \pm 5^{\circ}\text{F}$ ) prior to being subjected to pressure.

9.4 A fluid at the specified temperature must be circulated through all the test specimens for a minimum of one-half hour.

## 10 Test Procedure

10.1 Install three fitting connections, assembled per manufacturer's specifications, to the test system. Allow for a minimum of 30 cm (12 in.) of tube length between the test fitting connection and the free end closure.

10.2 The plumbing *must* permit free movement of each test specimen to allow for distortion.

10.3 Using the low pressure circulation system (see Section 7.1.2), condition the test specimens to 25°C (77°F) per 9.4. The system pressure is not to exceed 75 kPa (11 psig) during conditioning.

10.4 Properly position the isolation valves in such a way that only one specimen will be pressure tested, while the remaining specimens continue to have heated fluid circulating through them.

**WARNING: Take all necessary precautions when positioning the isolation valves, due to the potential harm which might be caused by an accidental exposure to the high temperature fluid.**

10.4.1 Test each specimen within two minutes after isolating it from the hot oil circulation system.

10.5 Using the high pressure system (see Section 7.1.1), pressurize the specimen until failure occurs.

10.5.1 Increase the pressure at a uniform and constant rate such that failure occurs between 60 to 70 seconds from initial exposure to the high pressure fluid.

10.6 Record on test data sheet (see Appendix 1 for sample test data sheet) the pressure and temperature at which the failure occurred. To identify mode of failure, record pressure reading under column:

“T” — for tube failure.

“F” — for fitting connection failure.

10.7 Repeat 10.1 through 10.5, substituting, for the fluid temperature in Section 10.2, the remaining six temperatures: 75°C (167°F), 100°C (212°F), 125°C (257°F), 150°C (302°F), 175°C (347°F), and 200°C (392°F).

## 11 Calculations

11.1 Calculate the average pressure for each test temperature and record in kPa (psig).

11.2 Calculate the standard deviation for each test temperature and record.

## 12 Data Accuracy

12.1 *Pressure* — kPa:  $\pm 2\%$

12.2 *Fluid Temperature* —  $\pm 1^\circ\text{C}$  ( $\pm 2^\circ\text{F}$ )

## 13 Test Data Sheet

The test data sheet shall include the following information:

13.1 Date tested.

13.2 Operator and test facility.

13.3 Description of items tested, including:

*Tubing* — manufacturer, O.D., wall thickness, part number, and material type.

*Fitting* — manufacturer, type, size, part number, and material type.

13.4 The actual temperature for each test.

13.5 The failure pressure for each sample, in the appropriate column.

13.6 The average and standard deviation for each failure mode of each specimen.

NOTE: A sample test data sheet is provided as Related Information 1.

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## RELATED INFORMATION 1

### SAMPLE TEST DATA SHEET

NOTE: This related information is not an official part of SEMI F10 and is not intended to modify or supercede the official standard. It has been derived from industry specifications. Publication was authorized by full letter ballot procedures. Determination of the suitability of the material is solely the responsibility of the user.

Test Date:	Operator:	Test Facility:
Tube Manufacturer:		Fitting Manufacturer:
Tube O.D.:		Fitting Tube:
Tube Wall Thickness:		Fitting Size:
Tube P/N:		Fitting P/N:
Tube Material:		Fitting Material:

Max. Pressure — kPa      T = Tube Failure, F = Fitting Connection Failure

<i>Actual Temperature</i>								
<i>Sample</i>	<i>25°C T</i>	<i>25°C F</i>	<i>75°C T</i>	<i>75°C F</i>	<i>100°C T</i>	<i>100°C F</i>	<i>125°C T</i>	<i>125°C F</i>
1								
2								
3								
Average =								
Standard Deviation =								

<i>Actual Temperature</i>						
<i>Sample</i>	<i>150°C T</i>	<i>150°C F</i>	<i>175°C T</i>	<i>175°C F</i>	<i>200°C T</i>	<i>200°C F</i>
1						
2						
3						
Average =						
Standard Deviation =						

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# **SEMI F11-0998**

## **TEST METHOD TO OBTAIN AN INDICATION OF THE THERMAL CHARACTERISTICS OF TUBE FITTING CONNECTIONS MADE OF FLUOROCARBON MATERIALS**

### **1 Purpose**

1.1 This method provides a uniform procedure to determine the thermal characteristics of tube fitting connections made of fluorocarbon materials.

### **2 Scope**

2.1 This method can be used to characterize tube fitting connections on the basis of test data developed under the conditions described herein, but the results are not intended to imply a performance rating.

2.2 Tube defined in this method has a circular cross section and is made of fluorocarbon materials.

2.3 Tube fittings defined in this method are made of fluorocarbon materials for all parts in contact with the internal fluid.

2.3.1 Parts such as a nut or gripper are not limited to being made of a fluorocarbon material.

2.4 Only the seal between the tube and tube fitting being evaluated is within the scope of this document. All other, threaded seals are beyond the scope of this document.

2.5 When using this method for making comparisons among various tube fittings and/or manufacturers, the user must be specific in the selection of the tube and tube fittings to be evaluated.

2.6 The International System of Units (SI) is used as the standard unit of measure in this document. The U.S. Customary units are in parentheses for reference purposes only and have been rounded to the nearest whole value.

### **3 Summary of Method**

Subject tube fitting connections made of fluorocarbon materials to extreme internal pressure and temperature conditions.

3.1 Test specimens are subjected to a constant internal pressure while controlling the increase of fluid temperature in uniform increments, with a cool down period between each elevated temperature condition.

3.2 After completion of the entire temperature range, test a new set of fittings and tubing with an increased pressure.

### **4 Significance and Use**

4.1 In the manufacturing of semiconductor products, many types of hazardous chemicals and solvents are required. As a result, tubing and various fitting designs made of fluorocarbon materials (which are chemically resistant to these fluids) are used to transport those fluids. It is important to control the testing process when evaluating various fitting designs, so that accurate comparisons of the thermal characteristics can be made.

4.2 It is the intent of this method to provide a procedure in which thermal transition testing of tube fitting connections made of fluorocarbon materials will be performed. By using this method, accurate comparisons of various tube fitting designs can be achieved.

4.3 The results obtained when using this method are applicable only to conditions that specifically duplicate the procedures used within this document.

4.4 When using this test method, it is assumed that the test specimens are truly representative of the material and manufacturing process specified for that product. Departure from this assumption could introduce discrepancies that are greater than those introduced by departure from the details of the procedure outlined in this method.

### **5 Terminology**

#### **5.1 Acronyms**

5.1.1 *O.D.* — Outside Diameter

5.1.2 *P/N* — Part number

#### **5.2 Definitions**

5.2.1 *characterize* — To describe the quality of.

5.2.2 *free end closure* — A metal tube fitting connection which is securely fastened to the tube and does not contribute to the restraint of the test specimen.

5.2.3 *subject* — To expose to.

5.2.4 *thermal transition* — A change from a specific elevated fluid temperature down to room temperature and then to an elevated temperature higher than previously tested, with the entire process repeated for multiple temperature conditions.

## 6 Description of Test Equipment

6.1 A hydraulic system capable of applying a constant internal hydraulic pressure to all test specimens while allowing a uniform fluid flow rate through each specimen.

6.1.1 The pressure capability shall range from 100 to 1200,  $\pm 35$  kPa (15 to 174,  $\pm 5$  psig).

6.1.2 The fluid flow rate shall be adequate to maintain the fluid temperature at each specimen to  $\pm 2.8^\circ\text{C}$  ( $\pm 5^\circ\text{F}$ ) of the specified fluid temperature.

6.1.3 The fluid temperature capability shall range from  $25^\circ\text{C}$  to  $200^\circ\text{C}$  ( $77^\circ\text{F}$  to  $392^\circ\text{F}$ ), within a one hour time interval from hot to cool and vice versa.

6.2 An enclosure that will allow observation of the test specimens and is capable of controlling the ambient temperature to  $31^\circ \pm 6^\circ\text{C}$  ( $88^\circ \pm 11^\circ\text{F}$ ).

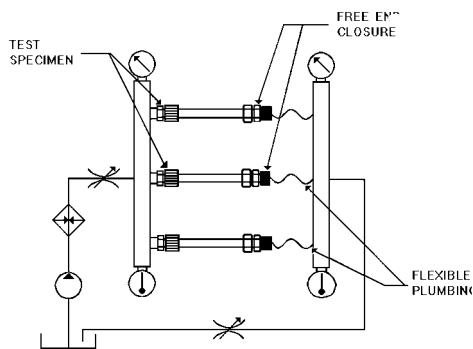
6.3 An instrument to monitor pressure within  $\pm 1\%$  of full scale.

6.4 An instrument to monitor temperature within  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

6.5 An instrument to monitor time.

6.6 *Specimen Support* — Any support is acceptable as long as it does not contribute to the restraint of the specimen in either the circumferential or axial direction.

6.7 See Figure 1 for basic test system.



**Figure 1**  
Basic Test System

## 7 Safety Precautions

**WARNING:** This test method will subject test specimens to conditions that may exceed the normal performance ratings of the products under evaluation. Adequate precautions must be taken to prevent injury to the person conducting the test. The hydraulic plumbing

and safety enclosure are to be constructed of materials which are capable of containing a fluid at  $200^\circ\text{C}$  ( $392^\circ\text{F}$ ).

## 8 Test Specimens and Conditioning

8.1 *Sample Size* — A minimum of three specimens shall be tested at each pressure condition.

8.2 *Specimen Surface* — All surfaces of the specimens shall be free of visible flaws, scratches, or other imperfections, unless typically found on a representative sample of the product.

8.3 *Specimen Conditioning* — All specimens must be conditioned for a minimum of one hour in an air environment of  $23^\circ \pm 2.8^\circ\text{C}$  ( $73^\circ \pm 5^\circ\text{F}$ ) prior to being subjected to pressure.

## 9 Test Procedure

9.1 Install three fitting connections, assembled per manufacturer's specifications, to the test manifold. Allow for a minimum of 30 cm (12 in) of tube length between the test fitting connection and free end closure. (See Figure 1.)

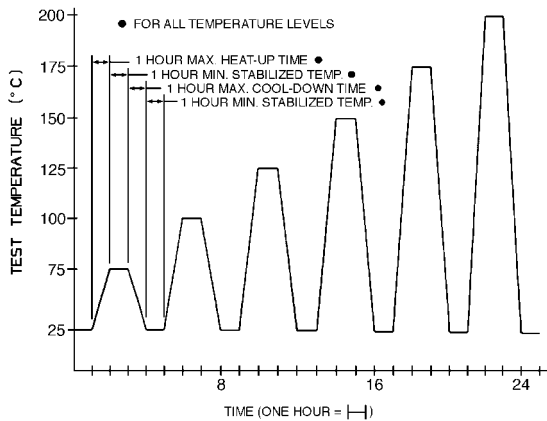
9.1.1 The apparatus should include a section of flexible line from the free end enclosure to return manifold to allow for expansion.

9.2 Circulate  $25^\circ\text{C}$  ( $77^\circ\text{F}$ ) fluid through the test samples with a constant pressure of 100 kPa (15 psig) for one hour.

9.2.1 When three failures occur at any given pressure during the initial room temperature condition (prior to the specimens having been subjected to any elevated temperature conditions), testing is terminated.

9.3 Increase the fluid temperature to  $75^\circ\text{C}$  ( $167^\circ\text{F}$ ) for a total time of two hours from the time the temperature was initially increased. The maximum time allowed to achieve the elevated temperature is one hour. (See Figure 2.)





**Figure 2**  
**Thermal Transition Chart**

9.4 Lower the fluid temperature to 25° C (77° F) for a total of two hours. The maximum time allowed to achieve the 25° C (77° F) temperature condition is one hour.

9.5 Repeat Sections 9.3 and 9.4, substituting, for the fluid temperature in Section 9.3, the remaining five temperatures: 100° C (212° F), 125° C (257° F), 150° C (302° F), 175° C (347° F), and 200° C (392° F).

9.5.1 The test samples should be at 25° C (77° F) prior to stopping the test at the end of a work shift.

9.5.1.1 The two hours at 25° C (77° F) will be equally divided between the present day cool-down time and the next day start-up time.

9.6 Monitor each of the three connections for external leakage throughout all test parameters.

9.7 Any external leakage at a tube connection will be recorded as a failure on the test data sheet (see Related Information 1 for sample test data sheet).

9.7.1 Leakage observed, once an elevated temperature is reached, will be recorded as a failure at that temperature.

9.7.2 Leakage at or during the cool-down cycle is to be recorded as a failure of the previously tested elevated temperature condition.

9.7.3 Leakage observed while increasing the temperature to the next condition is to be recorded as a failure of the previous elevated temperature condition.

9.8 Once a connection shows leakage and its failure pressure and temperature has been recorded, any additional leakage is not taken into consideration.

9.8.1 If leakage is not excessive, the connection can remain in the system until testing at that pressure has been completed.

9.8.2 If leakage of a connection becomes excessive, the connection may be retightened or replaced in order to continue testing of the remaining specimens. Take precautions not to disturb the remaining test specimens.

**WARNING: Due to the extreme danger of high temperature fluids, take all necessary precautions when retightening or replacing a connection, to prevent accidental exposure to the heated fluid.**

9.9 After three connection failures are recorded, or the 200° C (392° F) temperature has been achieved, a new set of fittings is to be installed for the next pressure condition.

9.10 Repeat Sections 9.2 through 9.9 for the remaining six pressures: 200 kPa (29 psig), 400 kPa (58 psig), 600 kPa (87 psig), 800 kPa (116 psig), 1000 kPa (145 psig), and 1200 kPa (174 psig).

## 10 Data Accuracy

10.1 Fluid Temperature —  $\pm 1^{\circ} \text{C}$  ( $\pm 2^{\circ} \text{F}$ )

10.2 Pressure — kPa:  $\pm 2\%$

## 11 Test Data Sheet

The test data sheet shall include the following information:

11.1 Date tested.

11.2 Operator and test facility.

11.3 Description of items tested, including:

*Tubing* — Manufacturer, O.D., wall thickness, part number, and material type.

*Fitting* — Manufacturer, type, size, part number, and material type.

11.4 The actual temperatures and pressures at which failures occur.

NOTE: A sample test data sheet is provided as Related Information 1.



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