

<i>Size of Pipe</i>	<i>"A" Not Exceeding</i>	<i>"A" Not Less than</i>
Less than 50 mm (2 in.)	6 mm (1/4 in.)	3 mm (1/8 in.)
50 mm to 75 mm (2–3 in.)	9 mm (3/8 in.)	3 mm (1/8 in.)
100 mm to 200 mm (4–8 in.)	13 mm (1/2 in.)	3 mm (1/8 in.)
250 mm and greater ( 10 in. and greater)	19 mm (3/4 in.)	3 mm (1/8 in.)

NOTE 1: Edge of hole must be clean and sharp or slightly rounded, free from burrs, wire edges or other irregularities. In no case shall fitting protrude inside pipe.

NOTE 2: Any suitable method of making the physical connection is acceptable if above recommendations are adhered to.

See ASME Performance Test Code PTC 19.5-1972, "Applications. Part II of Fluid Meters, Interim Supplement on Instruments and Apparatus.

**Figure 3**  
**Recommended Pressure Connection**



**Table 1 DUT Flow Pressure Drop for Integral Filtration Unit or Gas System**

DUT Unit Identification

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Test Number \_\_\_\_\_  
Date \_\_\_\_\_  
Operator Name \_\_\_\_\_  
Barometric Pressure \_\_\_\_\_  
Test Gas \_\_\_\_\_

Integral Filtration Unit or Gas System

	<i>Ambient Temperature (°C)</i>	<i>Inlet Pressure (psig, gauge)</i>	<i><math>\Delta P</math>(Unit) Differential Pressure, unit (psid, differential)</i>	<i>Flow Meter Reading (units)</i>	<i>Normal Flow Rate(m<sup>3</sup>/hr)</i>



**Table 2 Filter Flow Pressure Drop for Housing with Cartridge Element Removed**

Filter Unit Identification

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Test Number \_\_\_\_\_

Date \_\_\_\_\_

Operator Name \_\_\_\_\_

Barometric Pressure \_\_\_\_\_

Test Gas \_\_\_\_\_

Housing with Cartridge Removed

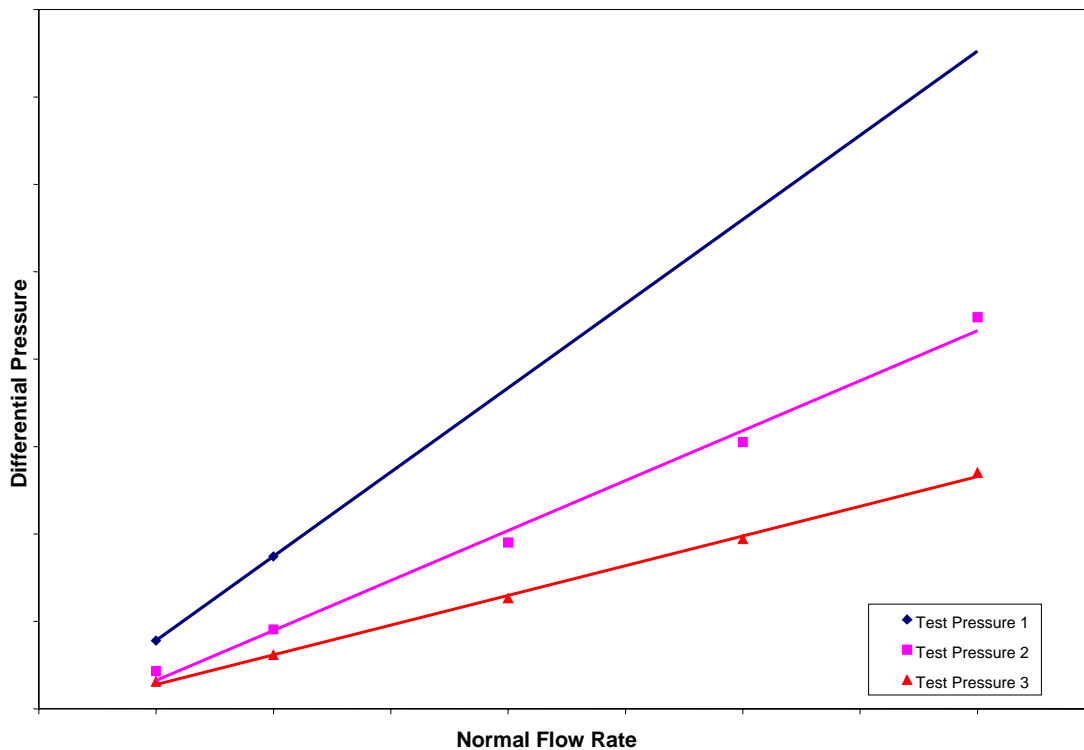
	<i>Ambient Temperature (°C)</i>	<i>Inlet Pressure (psig, gauge)</i>	<i>ΔP(Housing) Differential Pressure, Housing (psid, differential)</i>	<i>Flow Meter Reading (units)</i>	<i>Normal Flow Rate (m³/hr)</i>

**Table 3 Filter Flow Pressure Drop for Cartridge Element**

Cartridge Pressure Drop

$$\Delta P(\text{cartridge}) = \Delta P(\text{Unit}) - \Delta P(\text{Housing})$$

<i>Normal Flow Rate (m³/hr)</i>	<i>ΔP(Unit) Differential Pressure, unit (psid, differential)</i>	<i>ΔP(Housing) Differential Pressure, Housing (psid, differential)</i>	<i>ΔP(cartridge) Differential Pressure, Cartridge (psid, differential)</i>



**Figure 4**  
**Example of a Flow Pressure Drop Curve for a Filter**

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standard set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials 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 copy-righted 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 F60-0301**

## **TEST METHOD FOR ESCA EVALUATION OF SURFACE COMPOSITION OF WETTED SURFACES OF PASSIVATED 316L STAINLESS STEEL COMPONENTS**

This test method 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 November 22, 2000. Initially available at [www.semi.org](http://www.semi.org) December 2000; to be published March 2001.

### **1 Purpose**

1.1 The purpose of this document is to define a method for testing passivated 316L stainless steel components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable results among components tested for the purposes of qualification for this installation.

1.2 This document defines a method of testing the interior surfaces of stainless steel tubing, fittings, valves, and other components to determine the surface composition and chemistry, as a measure of the effectiveness of passivation processes.

1.3 The objective of this method is to describe a general set of instrument parameters and conditions that will achieve precise and reproducible measurements of important surface chemistry within the chromium-enriched oxide layer.

### **2 Scope**

2.1 This document describes a test method to characterize “as received” surface composition and chemistry encompassing all chromium-enriched stainless steel surfaces in tubing, fittings, valves, and other components. This procedure involves measurement of total Cr/Fe ratios,  $\text{Cr}^{\text{ox}}/\text{Fe}^{\text{ox}}$  oxide species ratios, and the surface elemental compositions by Electron Spectroscopy for Chemical Analysis (ESCA), also called X-ray Photoelectron Spectroscopy (XPS).

2.2 This document also describes the test method for a compositional ESCA depth profile measurement for Cr, Fe, Ni, O and C from the as-received surface, through the oxide layers, and extending into the base metal. The depth profile measurement evaluates the oxide thickness and the relative composition throughout the modified surface layer as a result of the passivation process.

2.3 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish

appropriate safety health practices and determine the applicability or regulatory limitations prior to use.

### **3 Limitations**

3.1 This test method is intended to be used by ESCA analysts familiar with the instrumentation and technique. The ESCA instrument must be calibrated and maintained to pertinent manufacturer’s specifications. The method is not intended to preclude the use of any particular brand or model of surface analysis equipment. While most of the test methodology has been developed using specific instrumentation, this method can be adapted to most surface analytical instrumentation.

3.2 The effects of the depth of analysis of the technique and surface contamination affect the results of this test method. These are discussed in the attached appendix. Surface roughness, non-planarity of the surface, and differential sputtering rates for the different chemical species also cause measurement uncertainties in this test method

### **4 Referenced Standards**

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

#### *4.1 SEMI Standards*

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

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

A276 — Standard Specification for Stainless Steel Bars and Shapes.

A751 — Standard Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products.

E673 — Standard Terminology Relating to Surface Analysis.

E902 — Standard Practice for Checking the Operating Characteristics of X-ray Photoelectron Spectrometers.

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<sup>1</sup> American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103

E1078 — Standard Guide for Specimen Handling in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy.

#### 4.3 NIST Standard<sup>2</sup>

SRD20 — NIST X-ray Photoelectron Spectroscopy Database.

#### 4.4 Physical Electronics<sup>3</sup>

Moulder, et al. — Handbook of X-ray Photoelectron Spectroscopy.

## 5 Terminology

### 5.1 Acronyms and Abbreviations

5.1.1 ESCA — Electron Spectroscopy for Chemical Analysis.

5.1.2 XPS — X-ray Photoelectron Spectroscopy, an equivalent name for ESCA.

### 5.2 Definitions

5.2.1 *sampling volume* — the volume from which photoelectrons are detected. The x-ray spot size and/or the lens and aperture system of the electron analyzer determine lateral dimensions. A length of three times the photoelectron mean free path is considered the maximum depth sensitivity. Sampling volume is dependent on the sample material and TOA. The acceptance angle of the analyzer will also influence the distribution of the depth information.

5.2.2 *take-off angle (TOA)* — the angle that the collection lens forms with the sample plane.

## 6 Summary of Method

### 6.1 Data Acquisition

6.1.1 Acquire elemental survey and calculate elemental composition of “as received” wetted surface.

6.1.2 Acquire chromium and iron regions at high resolution. Calculate total Cr/Fe ratio and Cr oxide/Fe oxide ratios from these regions.

6.1.3 Acquire a compositional depth profile to determine the relative abundance of C, O, Cr, Fe and Ni. Additional elements may be included as desired (i.e., molybdenum, silicon and nitrogen). If minor elements are included in the profile the instrument parameters should be chosen to allow for a detection limit of 1 atomic percent (at%) or better.

6.2 *Reporting* — Data is provided consisting of:

6.2.1 A survey spectrum extending from at least 0-1100eV.

6.2.2 The high-resolution Cr (2p<sub>3/2</sub>) and Fe (2p<sub>3/2</sub>) spectra from the as-received surface.

6.2.3 A compositional depth profile plot including C, O, Cr, Fe and Ni as a function of sputtering time.

## 7 Possible Interferences

7.1 *Mo (3d) and S (2s)* — use S (2p) for quantification of sulfur. Molybdenum will appear as a doublet; quantity may be overestimated.

7.2 *Fe (2p<sub>3/2</sub>) and Ni Auger* — ignore.

7.3 *Mo (3p) and N (1s)* — the ability to accurately quantify the nitrogen will depend upon the chemical state of both the nitrogen and molybdenum. In some cases the nitrogen will be overestimated from a survey scan quantification.

## 8 Apparatus

8.1 *Instrumentation* — Any ESCA instrument with definable area of analysis approximately 1 mm or less and sufficient count rate to provide detection of low level contaminants at one atomic percent or greater. A count rate of at least 100,000 counts per minute for clean Au (4f<sub>7/2</sub>) should indicate sufficient sensitivity.

8.2 The instrument should have peak resolution of 1 eV FWHM Au (4f<sub>7/2</sub>) or better (i.e., < 1 eV) to ensure species separation of metallic and oxide forms of chromium and iron.

8.3 If the instrument is of a variable take-off angle (TOA) design, the geometry should be set to a TOA of approximately 35°. Instruments with geometries significantly different from this, or with magnetic electron collection lens may provide analysis from a different sampling volume. The angle of the incident X-ray irradiation with respect to the sample plane and TOA must be recorded.

8.4 Data reduction software must have the ability to select an iterated Shirley-curve background.

## 9 Reagents and Materials

9.1 *Instrument Calibration Materials* — Refer to instrument manufacturer recommendations or ASTM E902. A measurement of the binding energy difference between the Au (4f<sub>7/2</sub>) at 84.0 eV and Cu (2p<sub>3/2</sub>) at 932.6 eV is an acceptable method of calibrating analyzer linearity. A value of  $\pm 3\sigma$  based upon a minimum of 10 historical measurements of less than  $\pm 0.25$  eV is acceptable.

<sup>2</sup> National Institute of Standards and Technology, Gaithersburg, MD 20899

<sup>3</sup> Physical Electronics, Eden Prairie, MN 55344

9.2 *Statistical Process Control Material* — A nominal 316L material with a composition range within the guidelines of ASTM A276. The composition of the bulk material should be determined by methods consistent with ASTM A751.

## 10 Safety Precautions

10.1 This test method does not purport to address the safety considerations associated with use of high voltage, vacuum, and X-ray producing equipment. The method assumes an ESCA analyst with knowledge of instrumentation and associated safety precautions.

## 11 Test Specimen

11.1 Specimens are to be sectioned to appropriate size for the particular ESCA instrument using a clean, dry hacksaw or dry low speed bandsaw. Any sample preparation shall avoid introducing contamination onto the surface to be measured. In addition, preparation must avoid excessive heating of the sample, i.e., the surface temperature shall not exceed 50°C, to avoid oxide growth or change in surface composition.

11.2 Sample preparation should preferably be done by the component manufacturer. Following sectioning the sample(s) are to be cleaned and packaged per the manufacturer's standard final cleaning and packaging procedures.

11.3 If sample preparation is done by other than the manufacturer, the sample(s) may be cleaned in DI water. If the sample(s) are not to be analyzed immediately they should be packaged by wrapping in clean metal foil or sealing in cleanroom quality nylon bags.

11.4 If sample preparation is done by other than the manufacturer this shall be stated in the report narrative and the analytical results are not to be interpreted as indicative of the manufacturer's quality of cleaning and packaging procedures. A note to this effect shall be included in all tables of reported results of the composition of the surface.

11.5 After preparation, samples should be analyzed promptly, with allowance for shipping times and queuing time at the analyst.

## 12 Preparation of Apparatus

12.1 Instruments shall be routinely tested in accordance with manufacturer recommendations to assure proper performance. The instrument vacuum shall be 1.0E-7 Torr or better during the analysis.

## 13 Calibration and Standardization

13.1 Proper instrument calibration is necessary to provide the most accurate and reproducible results, particularly if results are compared among different laboratories. Instrument calibration for sensitivity factors and binding energies shall be performed in accordance with the instrument manufacturer recommendations or other accepted method, such as that provided in ASTM E902.

13.2 The general ESCA calibration procedures described above are suitable for most analyses and provide reasonably accurate relative quantitative information regarding the surface chemistry. However, in order to establish the most accurate measurement practical and to maintain the desired level of reproducibility over time, an application-specific statistical process control (SPC) shall be established for this test method to determine the appropriate sensitivity factors for Cr, Fe, Ni and Mo in 316L stainless steel. The bulk composition of the SPC material should have an assay consistent with the acceptable composition range of 316L for Cr, Fe, Ni and Mo, as set forth in ASTM A276. The bulk composition of the SPC material should be determined by methods consistent with ASTM A751. The relative sensitivity factors established for the quantification of data should reflect the nominal composition of the bulk 316L SPC standard after a sputter cleaning sufficient to remove any surface contamination and the oxide passive layer. A  $\pm 3\sigma$  relative precision should be established from a minimum of 10 SPC measurements with the following tolerances:

Cr	$\pm 10\%$
Fe	$\pm 10\%$
Ni	$\pm 25\%$
Mo	$\pm 30\%$

As referenced from ASTM A276-97, a typical 316L material has a composition range in terms of wt% of:

Cr	16–18
Ni	10–14
Mo	2–3
C	< 0.03
Mn	< 2.0
P	< 0.045
S	< 0.03
Si	< 1.0
N	< 0.1
Fe	Balance.

Note that ESCA data is reported in atomic percent.

13.3 The sputter conditions established for the SPC surface cleaning shall be the same as those established for the ESCA depth profile measurement part of this

standard. A minimum sputtering etch cleaning of the SPC sample of  $\geq 10$  nanometers is recommended in order to remove surface contamination, the oxide passive layer and establish equilibrium. Note that the degree of preferential sputtering and other ion etching artifacts are a function of the matrix material and the ion gun conditions.

13.4 Calibration frequency of the application specific SPC should be designed to establish a record of the reproducibility and accuracy of the measurement, as reflected by the nominal composition of the 316L standard. A minimum frequency of one per week is recommended, or immediately prior to performing a measurement in accordance with this test method.

13.5 A sputter rate calibration shall be performed prior to an ESCA profile measurement conducted in accordance to this test method. The ion sputter rate determination shall be made using standard thin films of 100 nanometers or less of  $\text{SiO}_2$  on Si. The method of determining the thickness of the oxide film shall be based upon the oxygen concentration profile. The oxide – substrate interface shall be specified as the point at which the oxygen concentration decreases to  $\frac{1}{2}$  its maximum value in the  $\text{SiO}_2$  film, ignoring the first sputter cycle.

13.6 Multiple samples may be analyzed following a single sputter rate determination if the ion gun parameters have not been adjusted and the ion gun performance is documented as stable over the period in question.

## 14 Procedure

14.1 *As-Received Surface Analysis*— The sample is to be mounted in accordance with manufacturer's recommendations and in a manner consistent with ultra-high vacuum surface analytical procedures. Some of these practices are detailed in ASTM-E1078. The area to be analyzed should be mounted parallel to the sample holder surface so that TOA is well known.

14.1.1 Place the sample in the ESCA introduction chamber for pump down. Transfer to the analytical chamber at the manufacturer's recommended base pressure. Align the sample with respect to the X-ray beam and analyzer so that optimum count rate from the desired analytical location is obtained. The use of a collimated high voltage electron beam to align the sample should be avoided, as this may pyrolyze surface carbon and potentially alter surface oxide chemistry. The surface area to be analyzed should be free of visible particles and large defect features, if possible.

14.1.2 A beam size as close to 1 mm as possible should be used to ensure measurement of a representative surface. If surface curvature is great

(e.g.,  $< 1/4$ " tubing), a smaller beam may be employed. Elemental survey data (0–1100 eV) are to be measured from the sample surface to determine the elements present and their approximate surface abundance. A high throughput analyzer setting may be used to obtain a signal to noise ratio (S/N) sufficient to detect common surface contaminants such as sulfur and phosphorus at one atomic percent or better.

14.1.3 Using instrument settings sufficient to provide a FWHM peak width of 1.0 eV or less on Au ( $4f_{7/2}$ ), measure chromium, iron, and carbon regions. A typical region width is 20 eV. Suggested ranges are as follows: Cr ( $2p_{3/2}$ ) from 570 to 590 eV, Fe ( $2p_{3/2}$ ) from 700 to 720 eV, and C (1s) from 275 to 295 eV. Signal to noise ratios of greater than 20 are suggested.

14.1.4 A consistent method of data reduction of high-resolution surface spectra is necessary in order to provide meaningful comparison of the relative Cr:Fe ratios.

14.1.5 The spectra shall be charge compensated with respect to the maximum of the Cr ( $2p_{3/2}$ ) set to 577 eV. An iterated Shirley method of background subtraction shall be applied to the Fe ( $2p_{3/2}$ ) and Cr ( $2p_{3/2}$ ) spectral regions, using a minimum of three iterations.

14.1.6 The curve fit regions shall typically extend from:

Cr ( $2p_{3/2}$ )	570–582 eV
Fe ( $2p_{3/2}$ )	704–717 eV

14.1.7 The curve fit peak parameters should be initialized as follows:

Cr ( $2p_{3/2}$ )	Peak Position	FWHM	% gaussian
1	574	1.5	80
2	576.5	2.0	80
3	577.5	2.0	80
Fe ( $2p_{3/2}$ )	Peak Position	FWHM	% gaussian
1	707	1.0	80
2	708	1.0	80
3	710	1.4	80
4	711.5	1.4	80
5	713	1.4	80

14.1.8 The curve fit routine should apply the following tolerances for the band limit parameters of all peaks defined above:

FWHM delta	$\leq 0.2$ eV
% gaussian delta	$\leq 10\%$
Curve fit position delta	$\leq 0.25$ eV

14.1.9 An automated form of data reduction is recommended to enhance reproducibility and minimize



analyst subjectivity associated with the curve fit procedure. If it is necessary to deviate from the curve fit parameters outlined above in order to obtain a satisfactory curve fit it should be noted in the formal report. Possible sources for the anomalous behavior include an excessive concentration of iron oxide or a sufficiently aged sample surface, such that the oxide surface chemistry and structure has modified.

**14.2 Depth Profile Analysis—** The ESCA depth profile may be performed following acquisition of the initial as-received surface survey scan and high-resolution spectra using the same sample x-y position and take-off angle of 35°.

**14.2.1** The depth profile may be acquired in either continuous mode (simultaneous sputter etching and data acquisition) or alternating sputter etch/data acquisition mode. The following acquisition windows are recommended:

Spectral line	Lower limit	Range
C	280 eV	15 eV
O	525 eV	15 eV
Cr	570 eV	15 eV
Ni	848 eV	15 eV
Fe	700 eV	25 eV

**14.2.2** If only an elemental compositional profile is acquired, any pass energy may be selected as long as the remaining acquisition parameters defined by the number of sweeps, step size and dwell time result in ≤ 1.0at% detection of the matrix elements followed in the depth profile.

**14.2.3** The composition profile should extend far enough into the depth of the sample to reach the base metal composition of the 316L material, but a minimum of 100Å from the surface as referenced to the calibrated sputter rate in SiO<sub>2</sub>.

## 15 Calculations and Interpretation of Results

**15.1** Most manufacturers supply software for determination of elemental composition. The elemental composition should be calculated using sensitivity factors appropriate to the instrument, each element, and resolution settings for each measurement.

**15.2** The total Cr/Fe ratio is calculated by adding peak areas from all species of each element, adjusting for different numbers of scans and sensitivity factors, and

dividing the Cr result by the Fe result. The Cr oxide/Fe oxide ratio is calculated in a similar manner, except that only peaks 2 and 3 for Cr and only peaks 3 through 5 for Fe (oxide species) are used. The formulas are as follows:

$$\text{Total Cr/Fe} = \frac{(\sum \text{Cr peak areas})/\# \text{ Cr scans/Cr sensitivity factor}}{(\sum \text{Fe peak areas})/\# \text{ Fe scans/Fe sensitivity factor}}$$

$$\frac{\text{Cr Oxide/}}{\text{Fe Oxide}} = \frac{(\sum \text{Cr oxide peak areas})/\# \text{ Cr scans/Cr sens. factor}}{(\sum \text{Fe oxide peak areas})/\# \text{ Fe scans/Fe sens. factor}}$$

## 16 Reporting Results

**16.1 As-Received Surface Results —** A tabular summary of the elemental composition of all elements detected in the surface survey spectrum is to be supplied with the associated elemental survey spectrum. If sample preparation has been done by other than the component manufacturer all tables of as-received surface analysis results shall include a note stating: "Sample preparation was not performed by the component manufacturer. Results are not to be interpreted as indicative of the component manufacturer's quality of cleaning and packaging procedures."

**16.1.1** Tabular summaries of total Cr/Fe ratio and Cr oxide/Fe oxide are also to be supplied, with associated Cr(2p<sub>3/2</sub>), Fe(2p<sub>3/2</sub>), and C(1s) narrow region spectra.

**16.1.2** Acquisition parameters, including manufacturer, model and TOA; X-ray source (Al or Mg); beam size; and other pertinent settings are to be supplied. Analyst identity and analysis date are also required information. Each table and graph must be clearly labeled with sample identification.

**16.2 Depth Profile Results —** Compositional depth profile data plots shall display the atomic concentration of Fe, Cr, Ni, C, and O at minimum versus the sputter time or the equivalent depth from the calibrated sputter rate in SiO<sub>2</sub>.

**16.2.1** The method applied to determine the oxide thickness shall be specified in the report. A commonly accepted method of quantifying the oxide thickness is determined as the sputter time/depth at which the oxygen concentration decreases to ½ the maximum value. The carbon thickness may be determined similarly.

## APPENDIX 1

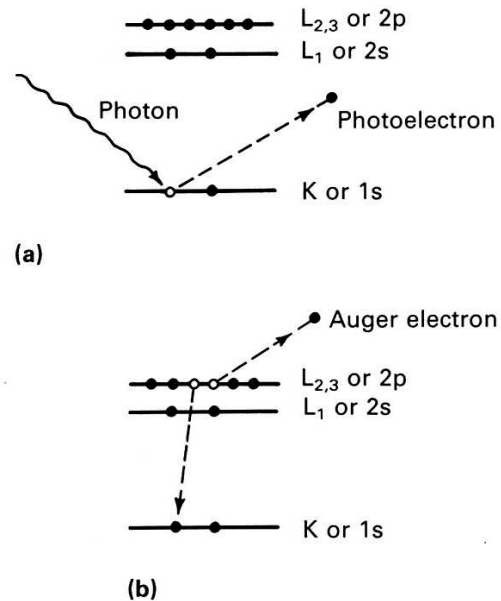
### DISCUSSION OF EFFECTS OF DEPTH OF ANALYSIS ON DEPTH PROFILE ANALYSIS BY ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS OF PASSIVATED STAINLESS STEEL

NOTE: This appendix is being balloted as an official part of SEMI F## by full letter ballot procedure, but the recommendation in this appendix are optional and are not required to conform to this standard. This appendix is derived from presentations made to the SEMI Surface Analysis Task Force.

A1-1.1 The purpose of this appendix is to describe the ESCA (sometimes referred to as X-Ray Photoelectron Spectroscopy (XPS)) technique and explain the interpretation of the depth profile analysis with respect to the structure and composition of the passive oxide layer on stainless steel. The effect of the depth of analysis on the measured chromium to iron ratio, the oxide thickness, and the depth of enrichment is discussed. The discussion applies to depth profile analyses performed by Auger electron spectroscopy, also.

A1-1.2 ESCA — The ESCA technique makes use of an X-ray beam for a probe. The surface of interest is illuminated with X-rays, typically Al  $K\alpha$  radiation, which has a well-defined energy of 1486.6 eV. When an x-ray photon and an electron interact, the electron adsorbs all of the energy of the photon, 1486.5 eV; it cannot react with it partially. The electron then has sufficient energy to leave the atom and move into the matrix. The electron's energy in the matrix is 1486.6 eV minus its binding energy to the atom from which it escaped. Its binding energy is indicative of the element or compound from which it came and the orbital from which it was removed. Such an electron is termed a "photoelectron".

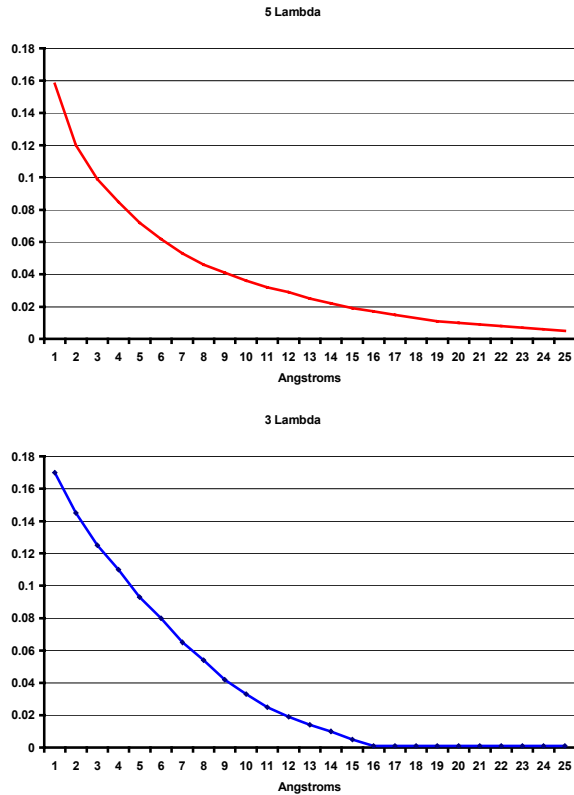
A1-1.3 In a typical ESCA instrument, a monochromatic beam of x-rays is focused at a point of interest on a surface. A cloud of electrons is generated at the surface by the x-rays. This electron cloud is made up of secondary electrons, Auger electrons and photoelectrons. (NOTE: The generation of Auger electrons is a byproduct of the process. If the photoelectron was an inner shell electron, when it leaves the atom it creates an ion in an excited state. Once the ion is created it can de-excite via the Auger process. The presence of Auger electrons in an ESCA survey can be used to determine what elements were present.) The mechanisms of ESCA photoelectron and Auger electron generation are illustrated in Figure A1-1.



**Figure A1-1**  
**Schematic representation of the ESCA photoelectron and Auger electron generation processes.**

A1-1.4 An electron lens is focused at the same point as the x-ray beam and a portion of the electron cloud enters the lens. From the lens, the electrons enter an electron analyzer that measures the number of electrons as a function of their kinetic energy. The kinetic energy is subtracted from the incident x-ray photon energy, and the resulting binding energy is then plotted as the x-axis with the number of electrons at each energy on the y-axis. An example is shown in Figure A1-2. The binding energy can be indicative not only of the element from which the electron came but also the chemical bonding state of the element.

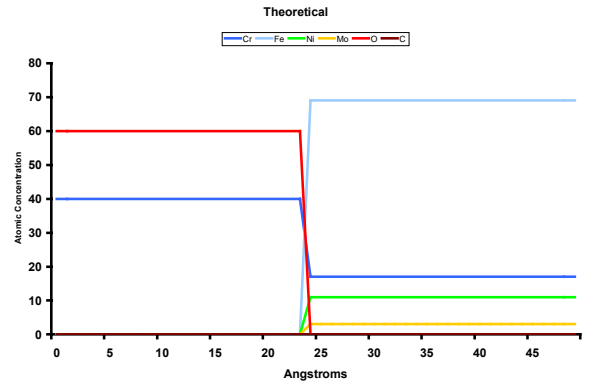




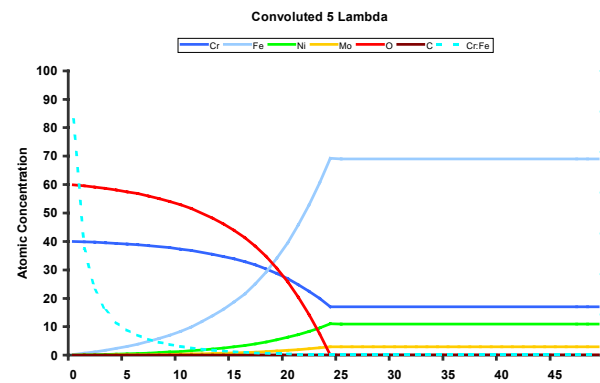
**Figure A1-4**

These two graphs represent the “Depth of Analysis” that will be seen when analyzing 1 keV electrons. The top curve is the  $5\lambda$  curve and bottom the  $3\lambda$ .

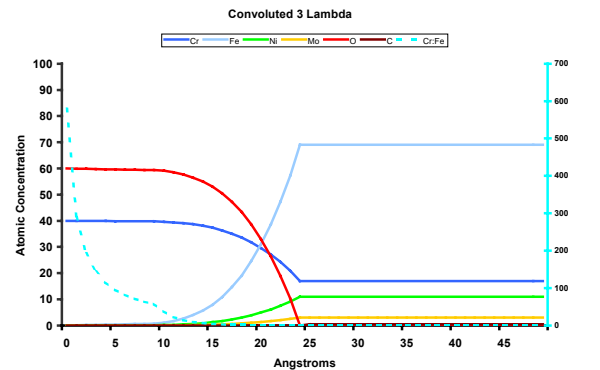
**A1-1.7 Modeling** — Figure A1-5 is the composition profile of a “perfect” oxide. It represents 25 Å of pure  $\text{Cr}_2\text{O}_3$  on an atomically flat 316L stainless steel surface, with no surface or interfacial contamination present. Figures A1-6a and A1-6b are the theoretical depth profiles derived by assuming a depth of analysis of  $5\lambda$  and  $3\lambda$  respectively. The first factor to note is that Fe, Ni and Mo appear most or all of the way through the depth profile of the oxide due to the contribution of the metal substrate to the detected signal, the “depth of analysis” effect as introduced above. 1 keV electrons can escape from as deep as 25 Å. This must be appreciated when interpreting these depth profiles.



**Figure A1-5**  
Composition versus depth of a pure  $\text{Cr}_2\text{O}_3$  layer on stainless steel.



**Figure A1-6a**  
Theoretical depth profile of Figure A1-5 assuming  $5\lambda$  depth of analysis.



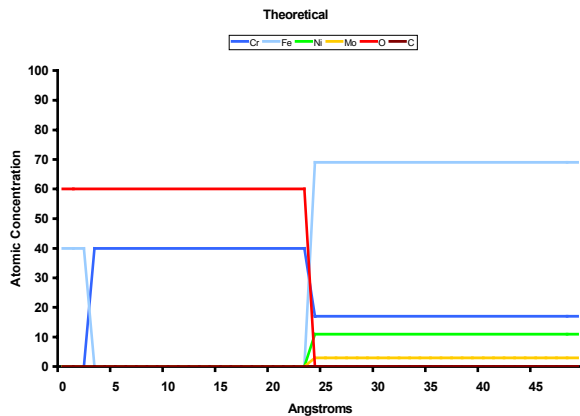
**Figure A1-6b**  
Theoretical depth profile of Figure A1-5 assuming  $3\lambda$  depth of analysis.

**A1-1.8** The second factor to notice is the thickness of the oxide. Historically, the FWHM of the oxygen profile has been used as the measure of oxide thickness.

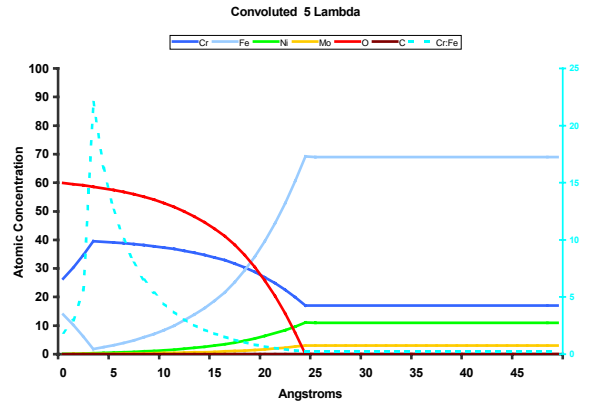
It is apparent in Figures A1-5 and A1-6 that this underestimates the oxide thickness by 15 to 20%.

A1-1.8.1 It should also be noted that the Cr:Fe ratio measured from Figures A1-5 and A1-6 has its maximum value at the initial surface, decreasing as the depth profile progresses, whereas the actual Cr:Fe ratio in Figure A1-5 is infinite down to 25 Å. Most actual depth profiles exhibit a maximum of the Cr:Fe ratio at some depth below the initial surface, referred to as the depth of maximum enrichment. This is a consequence of variation in the actual composition of the oxide, possibly having a higher concentration of Fe near the surface, a phenomenon commonly termed a “detached iron oxide layer”, or of contamination on the surface, generally hydrocarbons. These are discussed in the next section.

A1-1.9 *Effects of Detached Iron Oxide Layer and Surface Contamination* — The model composition profile of Figure A1-7 shows 3 Å of pure  $\text{Fe}_2\text{O}_3$  over 22 Å of pure  $\text{Cr}_2\text{O}_3$  on 316L stainless steel. This is a model of a detached iron oxide layer. Figure A1-8 is the theoretical depth profile of this model derived assuming a depth of analysis of 5 lambda. Note that the initial Cr value in the profile is higher than the Fe value, even though the surface is pure  $\text{Fe}_2\text{O}_3$ . This is a consequence of the depth of analysis detecting the Cr from levels beneath the surface. The Cr:Fe ratio for this model has its maximum value at 3 Å below the initial surface. The theoretical depth profile using a depth of analysis of  $3\lambda$  is very similar.

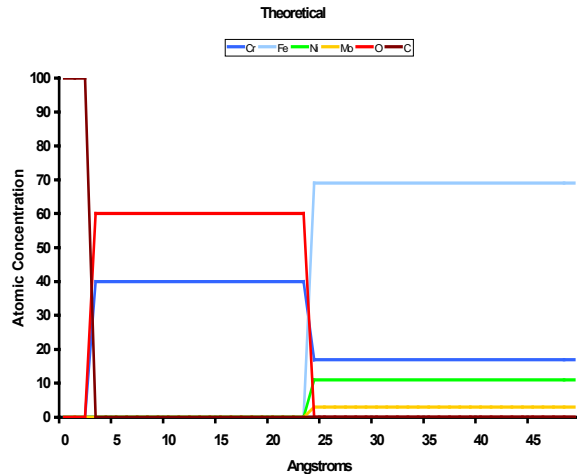


**Figure A1-7**  
Composition versus depth of a  $\text{Fe}_2\text{O}_3$  layer over a  $\text{Cr}_2\text{O}_3$  layer on stainless steel.

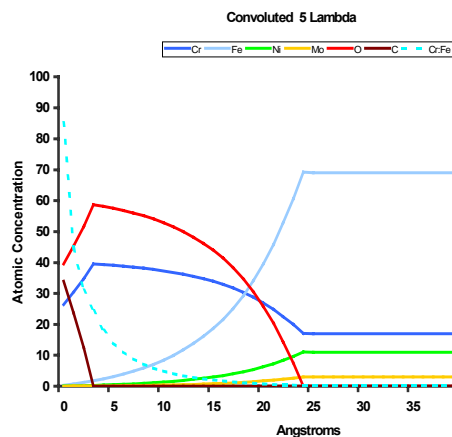


**Figure A1-8**  
Theoretical depth profile of Figure A1-7 assuming  $5\lambda$  depth of analysis.

A1-1.10 The model composition profile of Figure A1-9 shows 3 Å of pure carbon over 22 Å of pure  $\text{Cr}_2\text{O}_3$  on 316L stainless steel, representing an idealized model of the adsorbed hydrocarbon contamination generally found on stainless steel surfaces exposed to the atmosphere. Figure A1-10 is the theoretical depth profile of this model assuming a depth of analysis of  $5\lambda$ . In this case the O, Cr and Fe atomic concentration values are reduced by the presence of the carbon until the depth profiling proceeds past the carbon, but they have the same relative values (ie: same Cr:Fe ratio) versus depth as derived in the uncontaminated model. The maximum of the Cr:Fe ratio is seen to be at the initial surface. Although the actual oxide thickness is less in this model, the FWHM measure of the oxide thickness from the depth profile is the same due to the presence of the carbon layer. Note that the Oxygen concentration profile initially increases to a maximum, then decreases. This is typical of Oxygen concentration profiles seen on actual samples, which will generally have some adsorbed hydrocarbon contamination on the surface.

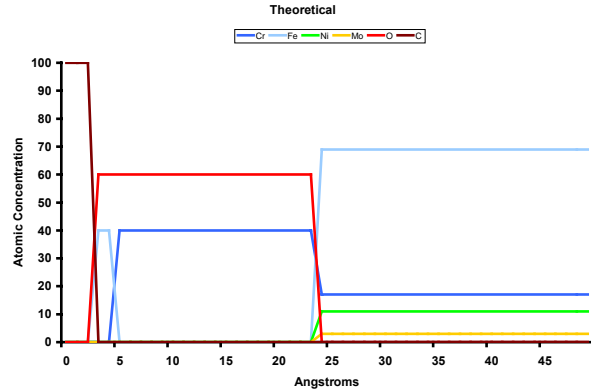


**Figure A1-9**  
Composition versus depth of a carbon layer over a  $\text{Cr}_2\text{O}_3$  layer on stainless steel.

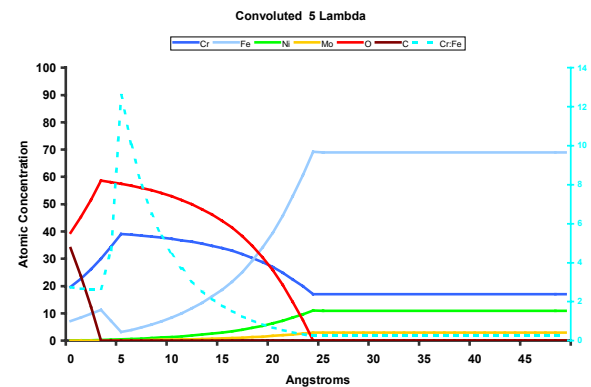


**Figure A1-10**  
Theoretical depth profile of Figure A1-9 assuming  $5\lambda$  depth of analysis.

A1-1.11 The model composition profile of Figure A1-11 shows a 3 angstrom carbon layer over 3 Å of pure  $\text{Fe}_2\text{O}_3$  over 19 Å of pure  $\text{Cr}_2\text{O}_3$  on 316L stainless steel. Figure A1-12 is the theoretical depth profile of this model assuming a depth of analysis of  $5\lambda$ . This profile is seen to be similar to the depth profiles generally observed for passivated stainless steel. The Cr:Fe ratio maximum occurs below the initial surface, and the initial surface atomic concentrations of the elements of interest are diluted by the presence of the carbon layer on the surface. The oxide thickness, as measured by the FWHM technique, is affected by both the presence of the carbon layer and the depth of analysis.



**Figure A1-11**  
Composition versus depth of a carbon layer over a  $\text{Fe}_2\text{O}_3$  layer over a  $\text{Cr}_2\text{O}_3$  layer on stainless steel.



**Figure A1-12**  
Theoretical depth profile of Figure A1-11 assuming  $5\lambda$  depth of analysis.

A1-1.12 It must be emphasized that these derived depth profiles are for models with perfect interfaces and perfect compositions instead of the compositional gradients observed in real systems. Additional measurement uncertainties result from roughness and non-planarity of the surface, and from differential sputtering rates for different chemical species during depth profiling.

A1-1.13 The depth profiles of real systems must be interpreted with an understanding of the effects described in this Appendix and a realization that they are not ideal. The same considerations pertain to Auger depth profile analysis.



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# SEMI F61-0301

## GUIDE FOR ULTRAPURE WATER SYSTEM USED IN SEMICONDUCTOR PROCESSING

This guide was technically approved by the Global Facilities Committee and is the direct responsibility of the North American Facilities Committee. Current edition approved by the North American Regional Standards Committee on November 22, 2000. Initially available at [www.semi.org](http://www.semi.org) December 2000; to be published March 2001.

### 1 Purpose

1.1 This guide establishes the typical definitional requirements for an ultrapure water (UPW) system used in semiconductor manufacturing. It is intended to establish a common basis for developing detailed specifications in subsequent documents concerning design, performance and certification and monitoring of UPW systems.

1.2 This document may be used by users and suppliers as a basis for developing site-specific UPW specifications and performance criteria.

### 2 Scope

2.1 This guide applies to ultrapure water systems used in semiconductor manufacturing facilities for supplying high purity water for chemical dilutions, wafer processing and other manufacturing processes.

2.2 This guide can be used to understand the design elements and functionality of all UPW systems, which includes a Reverse Osmosis (RO) and a Deionization (DI) process. However, it is most applicable to newer designed UPW systems that support submicron linewidth device manufacturing.

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

### 3 Limitations

3.1 This guide does not define the actual specifications generally negotiated between the user and the manufacturer of the UPW system, against which water samples are tested and qualification is passed.

3.2 This guide does not address the testing and prequalification of materials, subassemblies, or components used in a UPW system.

3.3 This guide does not address the protocols and requirements defined by the manufacturer concerning the installation of the UPW system.

3.4 This guide does not address the type, level, or frequency of testing necessary and appropriate for ongoing monitoring of a UPW system.

3.5 This guide does not address the frequency or scope of ongoing maintenance for UPW systems including change out of resin beds and replacement of filters.

3.6 This guide does not intend to cover any of the important safety considerations that relate to the proper installation, operation, or maintenance of a UPW system.

### 4 Terminology

#### 4.1 Acronyms and Abbreviations

4.1.1 *TOC* — Total organic carbon, also Total Oxidizable Carbon. Refers to organic compounds.

4.1.2 *UPW* — Ultrapure Water System consisting of multiple components including a Reverse Osmosis (RO) and a Deionization (DI) process.

#### 4.2 Definitions

4.2.1 *activated Carbon* — a media filter used to remove oxidizing agents, like chlorine and chloramines, and remove (adsorb) certain TOC compounds.

4.2.2 *anion* — a negatively charged ion.

4.2.3 *cation* — a positively charged ion.

4.2.4 *clarifier* — a piece of water treatment equipment, typically used at municipal drinking water plants, to remove suspended solids from surface water and/or to soften surface water.

4.2.5 *degasification* — the removal of a certain amount of volatile compounds dissolved in water.

4.2.6 *deionization (DI)* — the removal of undesirable ions from water.

4.2.7 *DI storage* — generally refers to a storage tank that contains DI water, located between the primary and polishing ion exchange subsystems.

4.2.8 *DI (deionized) water* — generally refers to water that has passed through a full-train ion exchange system or RO water that has been polished by ion exchange.



4.2.9 *dissolved solids* — contaminants in water that are so small that they are uniformly distributed, including ions and the smallest TOC and silica compounds.

4.2.10 *dual-beds* — an ion exchange scheme where a cation exchange unit is followed by an anion exchange unit.

4.2.11 *electrodeionization (EDI)* — a water treatment technology that utilizes mixed-bed ion exchange plus an electrical potential to remove undesirable dissolved solids. Also referred to in the industry as CDI (Continuous Deionization).

4.2.12 *filtration* — the removal of suspended solids by passing water through some form of solid or semi-solid medium.

4.2.13 *final filter* — generally the final treatment step in a UPW system; used to remove suspended solids.

4.2.14 *full-train DI* — an ion exchange scheme where a cation exchange unit is followed by an anion exchange unit and a mixed-bed ion exchange unit.

4.2.15 *ground water* — water located below the surface of the earth, also called *well water*.

4.2.16 *heat exchanger* — a piece of equipment used to control the temperature of a water stream.

4.2.17 *ion exchange* — a water treatment technology used in a high-purity water treatment application to exchange undesirable cations for hydrogen ions and undesirable anions for hydroxide ions.

4.2.18 *loop* — the distribution system that includes the continuous circulation of UPW from the Final Filter back to the DI storage tank. End users draw off of the loop.

4.2.19 *low-pressure UV units* — units that use UV lamps that have a slight vacuum within. Typically, low-pressure lamps are called *254 nm* for ozone destruction and bacterial inactivation or *185 nm* for TOC reduction.

4.2.20 *medium-pressure UV units* — units that use UV lamps that have a positive pressure within. Used with bacterial inactivation/ozone destruction lamps or TOC reduction lamps.

4.2.21 *microfiltration* — generally refers to filters designed to remove suspended solids less than one micron in size but greater than 0.1 micron in size.

4.2.22 *mixed-beds* — ion exchange vessels used to polish already purified water, in which both cation and anion exchange occurs.

4.2.23 *multimedia filter* — generally refers to a suspended-solids removal piece of equipment that

contains two or more filtering media such as anthracite and sand, or anthracite, sand and garnet.

4.2.24 *Ozone* — Ozone (O<sub>3</sub>) may be injected into the *Supply* and/or *Return* line to control microbiological contaminants and also to enhance the action of TOC breakdown in downstream TOC reducing UV units.

4.2.25 *polishing ion exchange* — a cation/anion exchange step located downstream of primary ion exchange.

4.2.26 *pretreated water* — generally refers to treated water that is fed to reverse osmosis (RO) units.

4.2.27 *primary ion exchange* — the first cation/anion exchange step in a high purity water treatment scheme.

4.2.28 *raw water* — any untreated natural water like river water, lake water, ground water, or seawater. May also refer to the treated feed water that enters a plant from a municipal drinking water source or other source.

4.2.29 *return* — the UPW sent to but not used by end users that returns to the DI storage tank.

4.2.30 *reverse osmosis (RO)* — a filtration technology that utilizes a semi-permeable membrane to remove essentially all suspended solids and the vast majority of all dissolved solids. Generally refers to water (permeate) that has passed through a reverse osmosis (RO) membrane.

4.2.31 *RO storage* — generally refers to a storage tank that contains RO water.

4.2.32 *scale inhibitor* — a chemical used to minimize or eliminate the precipitation of slightly-soluble salts, like calcium carbonate (limestone) or calcium sulfate (gypsum), within water treatment equipment.

4.2.33 *supply* — the UPW sent to end users.

4.2.34 *surface water* — water located on the surface of the earth, such as river water, lake water, and seawater.

4.2.35 *TOC* — total organic carbon, also Total Oxidizable Carbon. Refers to organic compounds.

4.2.36 *treated water* — water that has passed through water treatment equipment and/or received chemical injections in order to modify the dissolved and/or suspended solids content of the water.

4.2.37 *ultrafiltration* — generally refers to filters designed to remove all submicron suspended solids.

4.2.38 *ultraviolet (UV)* — electromagnetic energy with around a 100–400 nm (nanometer) wavelength.

4.2.39 *UPW (ultra pure water)* — the highest purity water produced by a semiconductor water treatment system, which is sent to the end users for use in manufacturing.

## 5 General Requirements

5.1 *Materials* — Components of the UPW system must be appropriate to the application and conform to electrical, mechanical, and purity requirements, as well as the corrosive properties of the UPW chemistry. These requirements are defined by the physical installation environment, local and national code interpretations, process requirements, and delivery specifications.

5.2 *System Installation* — The UPW system is installed according to a protocol that ensures mechanical integrity, leakproof operation, and no or minimal contamination being added from distribution throughout the system.

5.3 *Acceptance Tests* — Acceptance tests are conducted on each subsystem or system produced. Such tests may include performance demonstrations, demonstrations of reliability criteria, and achievement of purity standards. Such tests are the basis for acceptance or rejection by the purchaser against a pre-negotiated set of criteria for the performance of the system.

5.4 *Qualification Testing* — Qualification testing may include tests for resistivity, temperature, pressure, TOC, dissolved oxygen, particle levels, bacteria, total silica, dissolved silica, non-volatile residue, ions, and metals.

5.5 *Monitoring* — UPW systems are monitored for continuing performance against desired and achievable levels of quality. Action limits are generally set to determine when system performance suggests that corrective action is required.

5.6 *UPW specifications* — UPW systems are generally guaranteed to deliver a certain quality of water on an ongoing basis. The guaranteed performance is established in advance between the UPW equipment manufacturer and the system owner. Both qualification testing and monitoring testing use the guaranteed specifications to determine the parameters and levels of purity to be tested.

5.7 *Recycle/Reclaim Opportunities* — There can be several opportunities for using water within a UPW system, and should be recognized during the design phase when possible. Examples are; use of 1<sup>st</sup> pass reject for cooling tower make-up water, returning UF reject to Primary or Feed water Tank, re-use of last stage rinse waters as UPW for lower grade use areas,

i.e., CMP, isolation of CMP waste stream, for possible reclaim/reuse.

## 6 Source Water

6.1 *Raw Water* — is the raw material from which UPW is made. Untreated raw water is natural water that is obtained from a surface source such as a lake or river, or from a ground water source. The raw water to a UPW system is most frequently treated *Municipal Drinking Water*.

6.2 *Municipal Drinking Water* — Most natural raw waters must be treated in order to produce drinking water that meets federal and state requirements. There are upper limits for inorganic contaminants (e.g., asbestos, arsenic, copper, and lead), pesticides, volatile organic chemicals (e.g., benzene, trichloroethylene, toluene, and xylene), turbidity, microbiological contaminants, and radiological contaminants (e.g., radon 226, radon 228, tritium, and strontium 90). *Municipal Water Treatment* may utilize only chlorination, or filtration and chlorination for certain ground water sources. For many surface water sources, coagulation (injection of aluminum or iron salts), flocculation (injection of a long-chain polymer), sedimentation, lime or lime/soda ash softening, filtration, chlorination or chloramination (chlorine plus ammonia) and other steps may be required.

## 7 Major Treatment Processes

7.1 *Pretreatment* includes all of the water treatment steps ahead of the *Reverse Osmosis Membrane Treatment* step. These steps are primarily required to protect the membrane units from scaling with sparingly soluble salts, fouling with living or non-living suspended particles, or chemical attack by pH, oxidizing agents or other dissolved contaminants. Pretreatment equipment may include media filtration, micro filtration, or ultrafiltration (bulk suspended solids removal), 1–5 micron cartridge filtration (polishing step for suspended solids removal), sodium-cycle cation exchange (softening, to remove scale-forming cations), acid injection (to minimize cellulose acetate membrane damage and/or to control carbonate scales), scale inhibitor injection (to control scaling), activated carbon filtration (to remove oxidizing agents and certain organic molecules), and sulfite ion injection (to remove oxidizing agents).

7.2 *Reverse osmosis Membrane Treatment* provides extremely high rejection of dissolved ions (charged atoms and molecules), organic (carbon containing) compounds, silica (silicon containing) compounds, and virtually complete rejection of suspended contaminants, but will not reject dissolved gases and volatile organic compounds as well. Reverse osmosis (RO) units may

be configured in a *double-pass* arrangement consisting of two RO membrane units in series. *Permeate* (filtered water) from the first RO unit is sent to the second RO unit to be filtered again. It is not uncommon for the permeate from a double-pass RO unit to have a resistivity reading up to 0.5–4 Megohm-cm, with less than one milligram per liter of organic and silica contaminants. The more contaminants removed in the membrane treatment step, the lower the loading on the *Polishing* steps.

**7.3 Volatiles Removal** — The removal of dissolved oxygen, carbon dioxide, other gases, and volatile organic compounds is a necessary treatment step. The removal of these volatile contaminants is accomplished to acceptable levels in vacuum degasifiers and in membrane degasification units. The removal of volatiles that can form ions and the removal of volatile organic compounds reduce the loading on downstream *Polishing* equipment.

**7.4 Polishing** — The relatively low level of contaminants that were not removed in the *Membrane Treatment* and *Volatiles Removal* steps are polished down to acceptable levels in the *Polishing* steps. Ionic, organic, and silica contaminants are removed in typically two stages of *Ion Exchange* in series (primary beds and polishing beds). Organic compounds, measured as TOC (Total Organic Carbon, or Total Oxidizable Carbon), that are found downstream of the RO membrane units may be subjected to *TOC Reduction* ultraviolet (UV) irradiation (185 nanometer low pressure units or medium pressure units) to convert most of them into ionic compounds that can be effectively removed by *Primary Ion Exchange Units* (usually consisting of Separate Beds, Mixed Beds or EDI/CDI/E-Cell). TOC compounds that exit the primary ion exchange units are typically subjected to TOC reducing UV irradiation to break them into ionized compounds to be removed by the *Polishing Ion Exchange Units*. The vast majority of all living suspended particles (mainly bacteria) that enter any 254 nm UV unit are inactivated. Downstream filters with a pore size of less than or equal to 0.45 micron remove the inactivated bacterial bodies. The final filter prior to *Distribution* typically has a pore size of less than 0.2 micron.

## 8 Distribution

**8.1 Distribution** is frequently composed of one or more *Loops*. Each loop consists of UPW that continuously recirculates through appropriate piping from the final filters, to the manufacturing areas requiring UPW (end users), and back to a tank located within the *Polishing* section to be polished again. The UPW to the end-user area is commonly called the

*Supply*. The UPW that travels from the end-user area back to the water treatment area is commonly called the *Return*. Ozone may be injected into the *Supply* and/or *Return* line to control microbiological contaminants and also to enhance the action of TOC breakdown in downstream TOC reducing UV units. Sometimes ozone is injected into the *Supply* line for similar reasons (requires de-ozonation at the end-use points). Sometimes the UPW in the distribution loop is heated (hot loop) for particular processes.

## 8.2 Basic System Components

**8.3 General** — Each UPW system contains certain basic components and a variety of design options to meet particular customer and facility needs. An example of a UPW system is shown in Figure 1 attached.

## 9 Related Documents

### 9.1 SEMI Standards

SEMI F4 — Guide for Standard Performance, Practices, and Sub-Assembly for High Purity Piping Systems and Final Assembly for Semiconductor Manufacturing Equipment

SEMI F31 — Guide for Bulk Chemical Distribution Systems

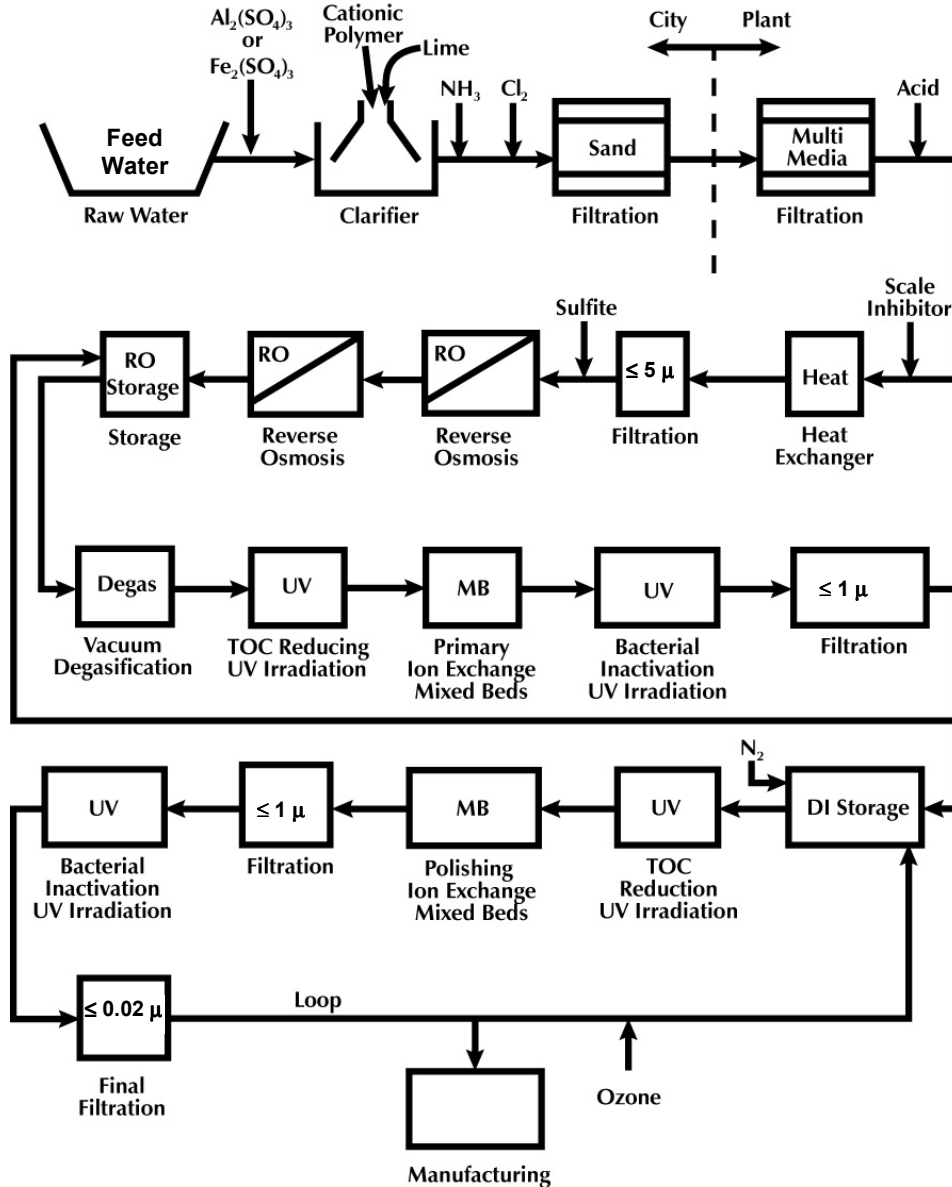
SEMI S2 — Environmental, Health, and Safety Guideline for Semiconductor Manufacturing Equipment

### 9.2 SIA<sup>1</sup>

National Technology Roadmap For Process Chemicals

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<sup>1</sup> Semiconductor Industry Association, 181 Metro Drive, Ste 150, San Jose, CA 95110, USA



**Figure 1**  
**Schematic of a Typical Ultrapure Water System**

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# SEMI F62-0701

## TEST METHOD FOR DETERMINING MASS FLOW CONTROLLER PERFORMANCE CHARACTERISTICS FROM AMBIENT AND GAS TEMPERATURE EFFECTS

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 Facilities Committee on March 22, 2001. Initially available at [www.semi.org](http://www.semi.org) May 2001; to be published July 2001.

### 1 Purpose

1.1 The purpose of this document is to define a method for testing MFCs being considered for installation into a gas distribution system and to quantify ambient and gas temperature effects on the MFC's indicated and actual flow.

### 2 Scope

2.1 This test method applies to metal and polymer sealed MFCs with flow rates up to 30 slpm. The tests include those listed below and are to be performed in the following order:

1. Ambient Temperature Effects (Steady State and Transient)
2. Gas Temperature Effects (Steady State and Transient)

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

### 3 Limitations

3.1 This method evaluates mass flow controllers in typically encountered, realistic operating conditions.

3.2 This test method does not address operational influences outside of the manufacturer's published limitations.

### 4 Referenced Standards

4.1 None.

### 5 Terminology

#### 5.1 Acronyms and Abbreviations

- 5.1.1 *DUT* — device under test
- 5.1.2 *g* — gravity
- 5.1.3 *kPa* — kiloPascal
- 5.1.4 *MFC* — mass flow controller

5.1.5 *NIST* — National Institute of Standard Technologies

5.1.6 *sccm* — standard cubic centimeters per minute

5.1.7 *slpm* — standard liters per minute

#### 5.2 Definitions

5.2.1 *stability, long term* — reading  $\pm 2\%$  for over one hour.

5.2.2 *stability, short term* — reading  $\pm 2\%$  within five minutes.

5.2.3 *zero setpoint* — manufacturer's setpoint for no flow from the MFC.

#### 5.3 Descriptions of Terms

5.3.1 *MFC<sub>I</sub>* — inlet mass flow controller.

5.3.2 *T<sub>a</sub>* — the temperature in the environmental control chamber.

5.3.3 *T<sub>g</sub>* — gas temperature at the inlet to the DUT.

5.3.4 *T<sub>max</sub>* — maximum manufacturer-rated temperature for an MFC.

5.3.5 *T<sub>min</sub>* — 20°C or manufacturer rated minimum temperature, whichever is greater.

5.3.6 *TC<sub>g</sub>* — temperature coefficient due to changing gas temperature of DUT.

5.3.7 *TC<sub>e</sub>* — temperature coefficient due to changing environmental temperature of DUT.

5.3.8 *Q<sub>a</sub>* — for the purpose of this method, the output value of the flow standard in units of mass flow.

5.3.9 *Q<sub>ind</sub>* — for the purpose of this method, the output value of the device under test in units of mass flow.

5.3.10 *Q<sub>sp</sub>* — for the purpose of this method, the intended output in mass flow units for a particular setpoint.

5.3.11 *V<sub>a</sub>* — valve A

5.3.12 *V<sub>b</sub>* — valve B

5.3.13 *V<sub>Ii</sub>* — inlet isolation valve

5.3.14  $V_{io}$  — outlet isolation valve

## 6 Significance and Use

6.1 This test method defines a procedure for testing components being considered for installation in a gas distribution system. Application of this method is expected to yield data allowing an end user to choose among components tested for the purpose of qualification for this installation.

## 7 Apparatus

7.1 See Figures 1a and 1b for schematics of the set-ups.

7.2 *Flow Measurement Device*, National Institute of Standard Technologies (NIST) traceable and capable of measuring steady-state and transient flow characteristics, preferably with differential pressure sensors across a flow restriction. This device should have a relative accuracy of at least 2 to 1 with respect to the DUT.

7.3 *Temperature Measurement Device*, to measure test gas temperature, NIST traceable and capable of real-time measurement, with a low mass ( $< 0.05$  g) sensor and with low thermal losses through the leads ( $< 0.3$  mW/°C).

7.4 *Pressure Measurement Device*, to measure test gas pressure, NIST traceable, and capable of real-time measurement to measure test gas pressure.

7.5 *Gas Pressure Control Regulators*, to maintain test gas pressure at  $275 \text{ kPa} \pm 5 \text{ kPa}$ .

7.6 *Gas Temperature Control*, heat exchangers and heaters to maintain the gas temperature equal to the test temperature within  $\pm 1^\circ\text{C}$  at the test flow rates.

7.7 *Environmental Chamber*, capable of stable temperature control ( $\pm 1^\circ\text{C}$ ) from at least  $T_{\min}$  to a point exceeding the maximum rated MFC temperature by  $10^\circ\text{C}$ .

## 8 Reagents and Materials

8.1 *Test Gas*, clean, dry nitrogen.

8.2 *Tubing*, cleaned and maintained to have no adverse effects on the test.

8.3 *Valves*, capable of unimpaired operation at  $100^\circ\text{C}$ .

## 9 Safety Precautions

9.1 All manufacturers' recommendations should be followed and noted when testing the unit. Any safety precautions should always be followed.

9.2 This standard does not purport to address all of the safety problems, if any associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations before using this method.

## 10 Technical Precautions

10.1 The flow standard may read a non-zero value due to heating of the gas in the test to determine transient effects on the zero. These readings should be noted.

## 11 Preparation of Apparatus

11.1 *Setup and Schematic*, (see Figure 1a) *Ambient Temperature Effects*

11.1.1 Install an ambient temperature sensing device within 5–10 centimeters horizontally adjacent to the MFC body. The probe should not be in contact with the MFC body or the environmental chamber walls.

11.1.2 Position the gas temperature sensor in the center of the gas stream no more than 10 cm upstream from the inlet surface of the MFC body.

11.1.3 After installation of the MFC, flow nitrogen at a 100% setpoint to purge the system for a sufficient time to remove atmospheric contamination.

11.1.4 If the MFC has a selectable Auto Zero function, note this on all data presentation documents before proceeding with the test.

11.1.5 Minimize the length of tubing between the heater and the device under test to reduce gas temperature changes.

11.2 *Setup and Schematic*, (see Figure 1b) *Gas Temperature Effects*

11.2.1 Install a base temperature sensing device,  $T_c$  adjacent to the MFC body. The probe should be in contact with the MFC body.

11.2.2 Install an ambient temperature sensing device within 5–10 centimeters horizontally adjacent to the MFC body. The probe should not be in contact with the MFC body or the environmental chamber walls.

11.2.3 Position the gas temperature sensor in the center of the gas stream no more than 10 cm upstream from the inlet surface of the MFC body.

11.2.4 After installation of the MFC, flow nitrogen at a 100% setpoint to purge the system for a sufficient time to remove atmospheric contamination.

11.2.5 If the MFC has a selectable Auto Zero function, note this on all data presentation documents before proceeding with the test.

## 12 Calibration and Standardization

12.1 The flow standard, temperature standards, and pressure standards used are to be NIST traceable.

12.2 All ancillary equipment must be calibrated and maintained to the manufacturer's recommendations. Current calibration records must be maintained.

## 13 Conditioning

13.1 Reference conditions as listed in Appendix 1 are to be maintained unless otherwise noted.

## 14 Procedure

### 14.1 Environmental Temperature Effect

14.1.1 Install the test specimen in the test set-up according to the manufacturer's recommendations, see Figure 1a.

14.1.2 Set the environmental chamber temperature ( $T_e$ ) to  $T_{min}$  and allow it to stabilize for one hour. Ensure that gas temperature ( $T_g$ ) and ambient temperature ( $T_a$ ) are within 2°C of each other before beginning data collection.

14.1.3 Close  $V_{ii}$  and  $V_{io}$ , and record the MFC  $Q_{ind}$  at a zero setpoint.

14.1.4 Open  $V_{ii}$  and  $V_{io}$ . Change the MFC flow setpoints to 25%, 50%, 75%, and 100% of full rated scale.

NOTE 1:  $T_g$  and  $T_e$  must be within 2°C of each other.

14.1.5 At each setpoint, record  $T_g$ ,  $Q_{ind}$  once, and a minimum of 10 values at a maximum of five second intervals of  $T_e$  and  $Q_a$  for at least 60 seconds. Record the average of the 10 values as shown in Table 3.

14.1.6 Repeat Section 14.1.3.

14.1.7 Maintain the setpoint at zero and change the environmental chamber temperature  $T_{em}$  to the next level indicated in Figure 2.

14.1.8 Record the  $Q_{ind}$  at the zero setpoint in real time (max 30-second intervals) as the temperature  $T_e$  is changing. See example of data collection shown in Table 4.

14.1.9 Allow temperature  $T_e$  to stabilize for a minimum of one hour.

NOTE 2: The time for the  $T_e$  to stabilize at each level should be increased to two hours if the indicated flow at  $T_e$  level 1 and 9, or 2 and 8, or 3 and 7, or 4 and 6 are not within 5% of each other. See Figure 2.

14.1.10 Open the isolation valves  $V_{ii}$  and  $V_{io}$ .

14.1.11 Repeat Sections 14.1.3–14.1.9 for each  $T_e$  level, as shown in Figure 2.

### 14.2 Gas Temperature Effect

14.2.1 Install the test specimen in the test set-up according to the manufacturer's recommendations, see Figure 1b.

14.2.2 Allow the system to stabilize for one hour. See Figure 1. Refer to Figure 4 for flow chart.

14.2.3 Set the heater temperature to raise the gas temperature,  $T_g$ , such that  $T_g$  is elevated 10°C above ambient.

14.2.4 Simultaneously open  $V_{ii}$  and close  $V_{io}$ . Continue to monitor  $T_g$ ,  $T_e$ , indicated flow, and actual flow for one hour. If after one hour, the actual flow or  $T_g$  has not achieved long-term stability, or if  $T_g$  is not equal to  $T_e + 10$  ( $\pm 2^\circ\text{C}$ ), continue until these conditions are met or until an additional one hour period has elapsed. If long-term stability is not achieved, note this occurrence and record the average value of 10 samples minimum taken at maximum five-second intervals for each of  $T_g$ ,  $T_e$ , indicated flow, and actual flow as shown in Table 5, for at least 60 seconds.

NOTE 3: If  $T_g$  is not 10°C above ambient return to 14.2.3 and adjust the temperature.

14.2.5 Repeat 14.2.3–14.2.4 until all required setpoints have been run.

## 15 Calculations or Interpretation of Results

### 15.1 Calculations

15.1.1 The measured values for  $Q_{ind}$ ,  $Q_a$ , and  $T_g$  are determined by an arithmetic average of samples taken at time intervals after stability has been achieved at each temperature and flow condition.

15.1.2 The ambient temperature coefficient of flow shall be calculated as follows:

$$TC_e (\% / ^\circ\text{C}) = \frac{Q_{a2} - Q_{a1}}{Q_{sp} (T_{e2} - T_{e1})} \times 100$$

where  $Q_{a1}$  and  $Q_{a2}$  are the actual flow rates measured at ambient temperatures  $T_{e1}$  and  $T_{e2}$  respectively. For zero setpoint use the following.

$$TC_{zero} = \frac{Q_{a2} - Q_{a1}}{Q_{fullscale} (T_{g2} - T_{g1})} \times 100$$

This yields a result expressed as a percent of reading per °C. See example of data collection and analysis shown in Table 3. Final  $TC_e$  is to be presented in Table 2 format.

15.1.3 The gas temperature coefficient of flow is to be calculated as follows:

$$TC_g (\%/^{\circ}C) = \frac{Q_{a2} - Q_{a1}}{Q_{sp}} \times (T_{g2} - T_{g1}) \times 100$$

where  $Q_{a1}$  and  $Q_{a2}$  are the actual flow rates measured at gas temperatures  $T_{g1}$  and  $T_{g2}$  respectively. For zero setpoint use the following.

$$TC_{zero} = \frac{Q_{a2} - Q_{a1}}{Q_{fullscale}} \times (T_{g2} - T_{g1}) \times 100$$

This yields a result expressed as a percent of reading per  $^{\circ}C$ . Refer to Table 5 for an example of data collection and analysis. Final TCg is to be presented in Table 1 format.

## 16 Reporting Results

NOTE 4: All special features (remote electronics, auto zero, etc.) should be noted on the testing results for comparative analysis.

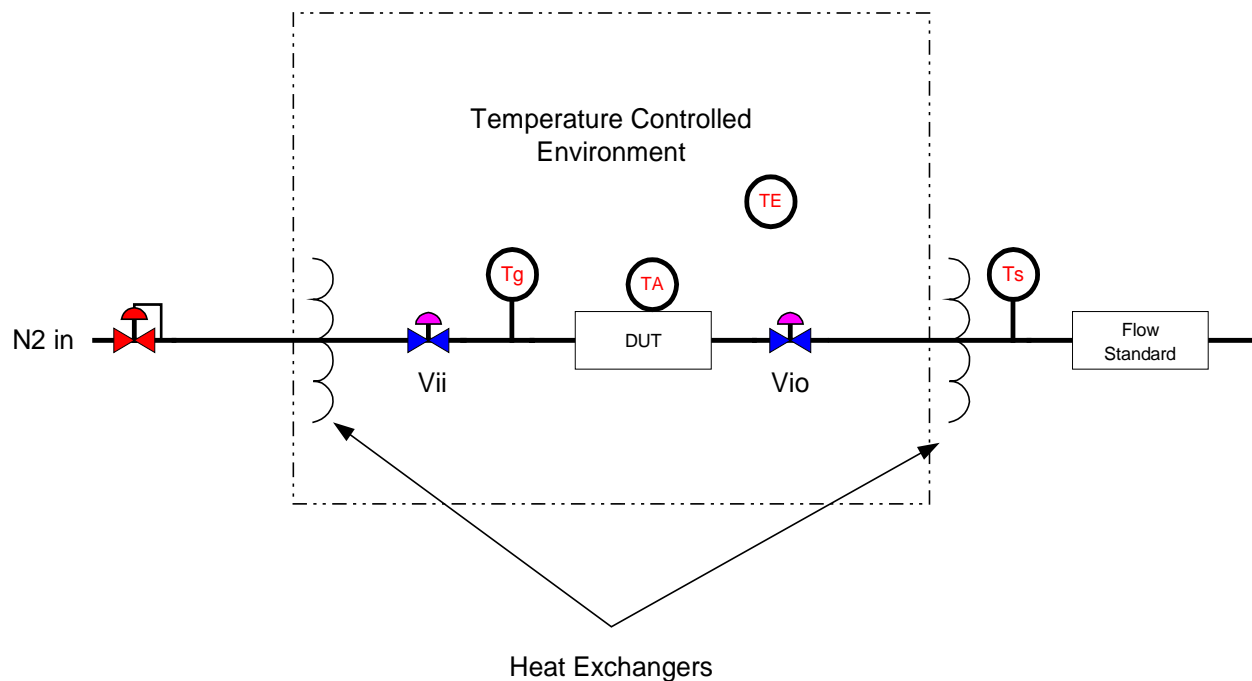
16.1 The gas temperature coefficient at each flow rate is to be displayed as shown in Table 1.

16.2 The gas temperature coefficient data from Table 1 is to be displayed as shown in Figure 5. The gas temperature, actual flow, and indicated flow are to be displayed versus time as shown in Figure 6.

16.3 The ambient temperature coefficient is to be presented in tabular form as shown in Table 2.

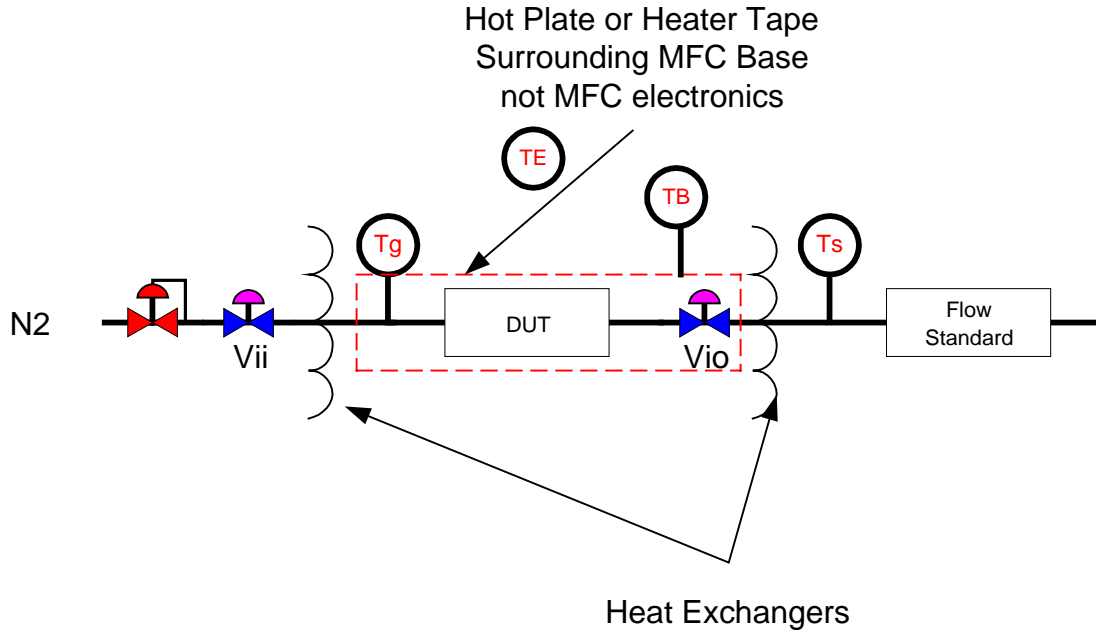
16.4 The data for the steady-state effect from Table 2 should be presented as shown in Figure 7. The data for the transient effect from Table 4 should be presented as shown in Figure 8.

## 17 Illustrations



**Figure 1a**  
**Ambient Temperature Effect Test Apparatus**



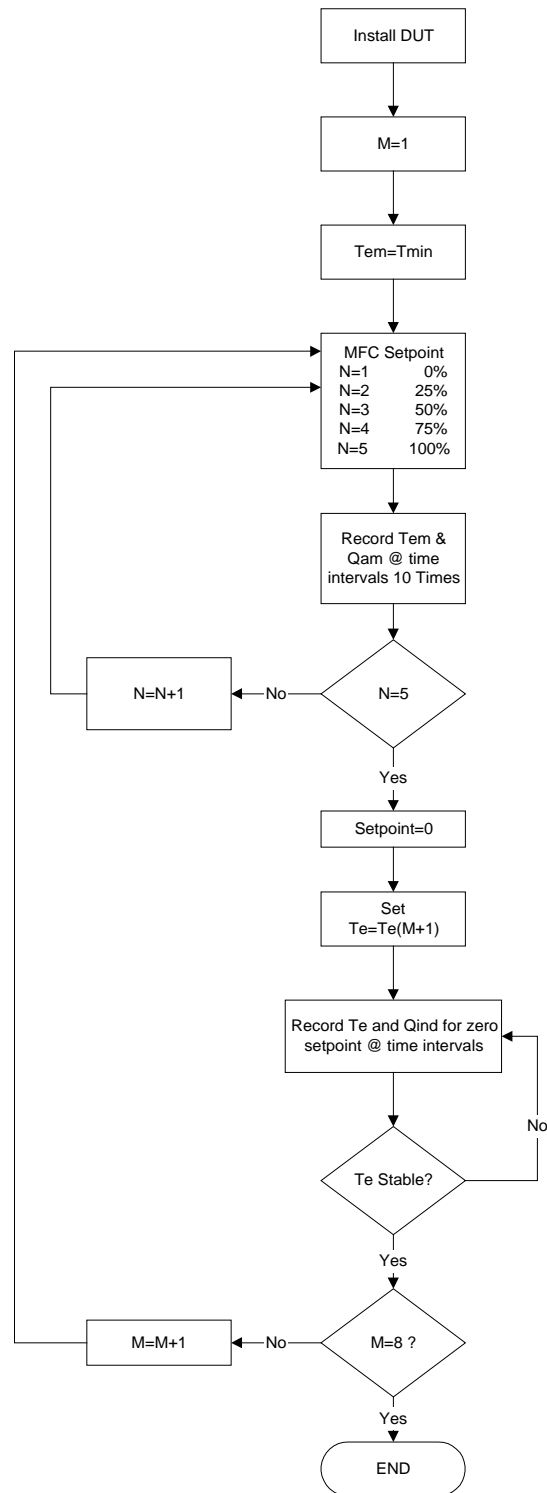


**Figure 1b**  
**Gas Temperature Effect Test Apparatus**

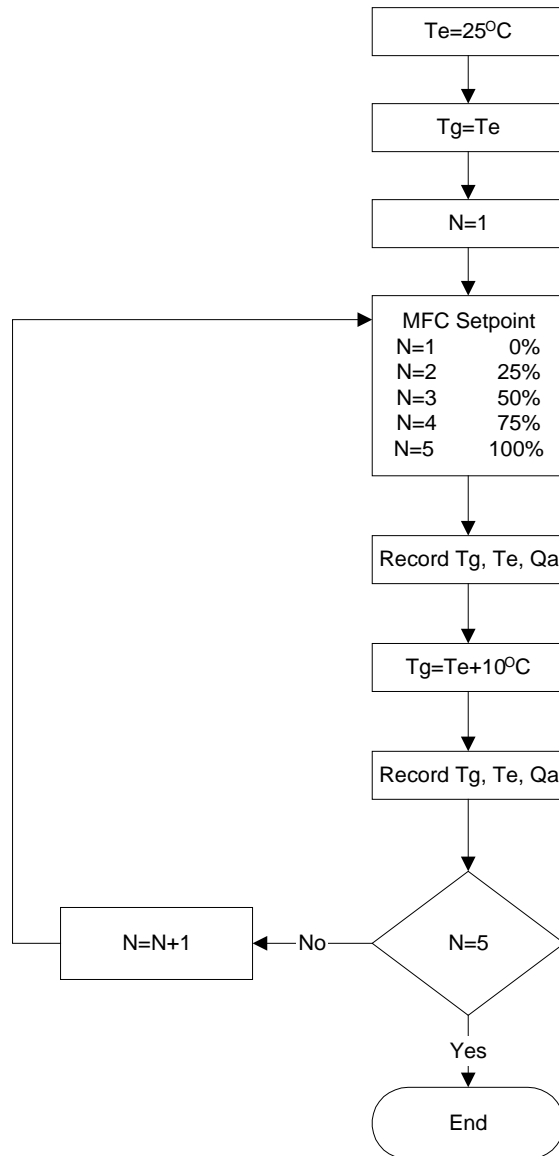
NOTE 5: Flow Standard may be located upstream or down stream.

Step m	$T_{em}$
1.	$T_{min}$
2.	$T_{min} + 1/4 (T_{max} - T_{min})$
3.	$T_{min} + 1/2 (T_{max} - T_{min})$
4.	$T_{min} + 3/4 (T_{max} - T_{min})$
5.	$T_{max}$
6.	$T_{min} + 3/4 (T_{max} - T_{min})$
7.	$T_{min} + 1/2 (T_{max} - T_{min})$
8.	$T_{min} + 1/4 (T_{max} - T_{min})$
9.	$T_{min}$

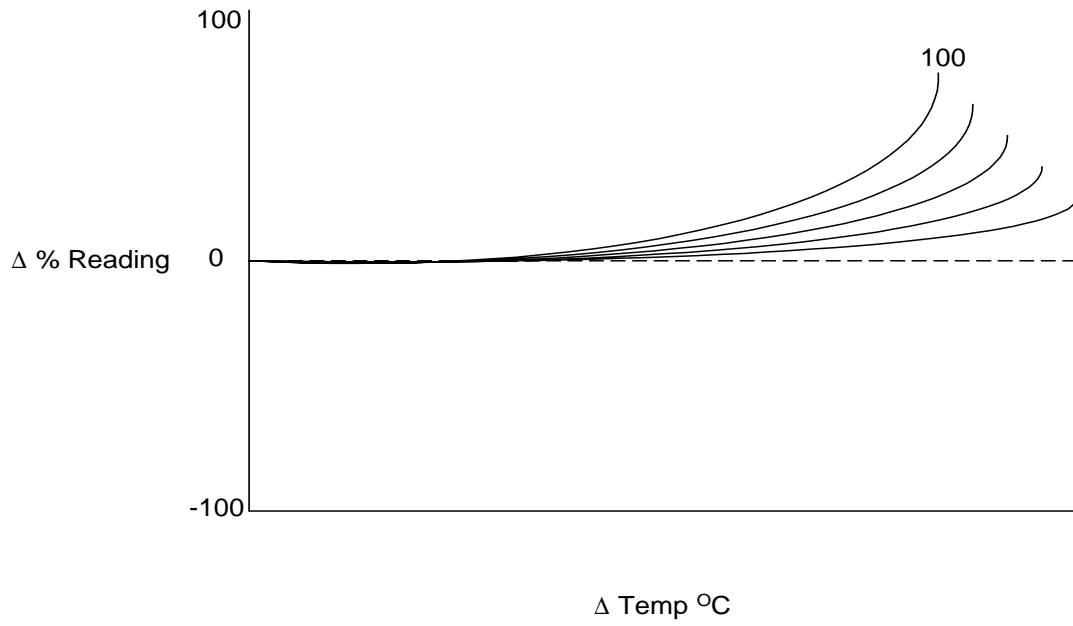
**Figure 2**  
**Ambient Temperature Setpoint ( $T_{em}$ )**



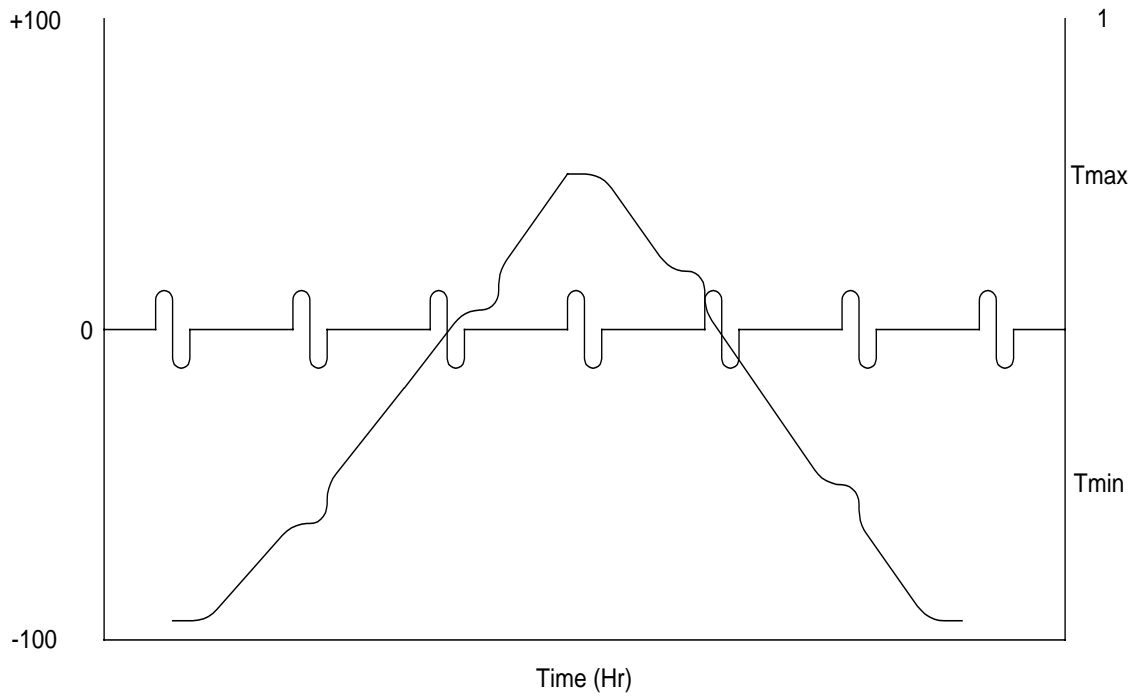
**Figure 3**  
**Ambient Temperature Test Procedure Flow Chart**



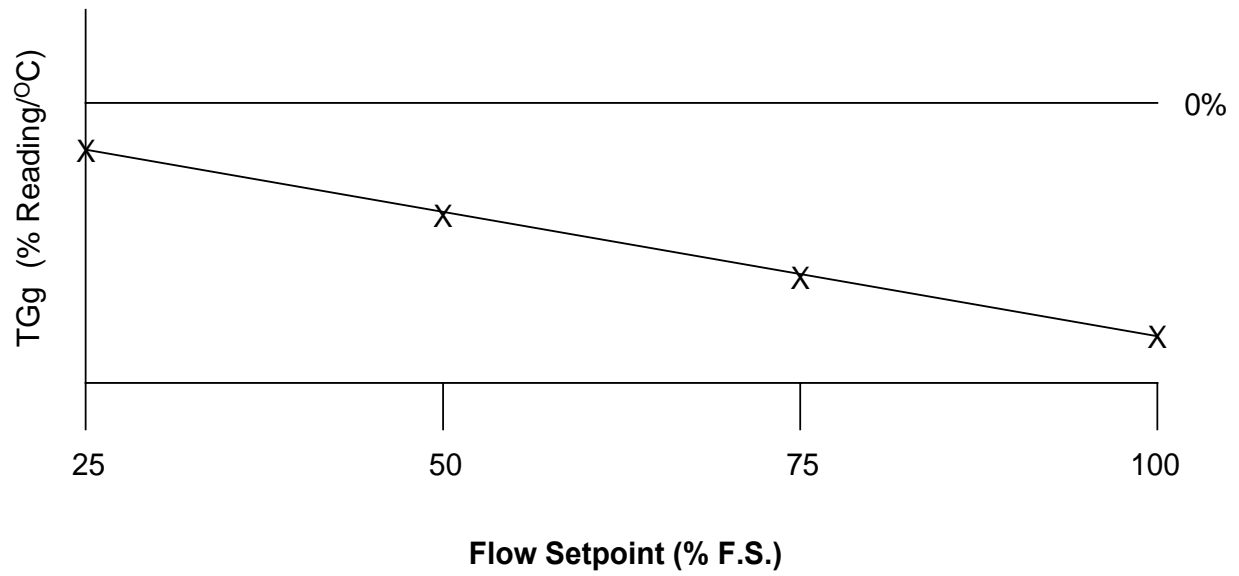
**Figure 4**  
**Gas Temperature Test Procedure Flow Chart**



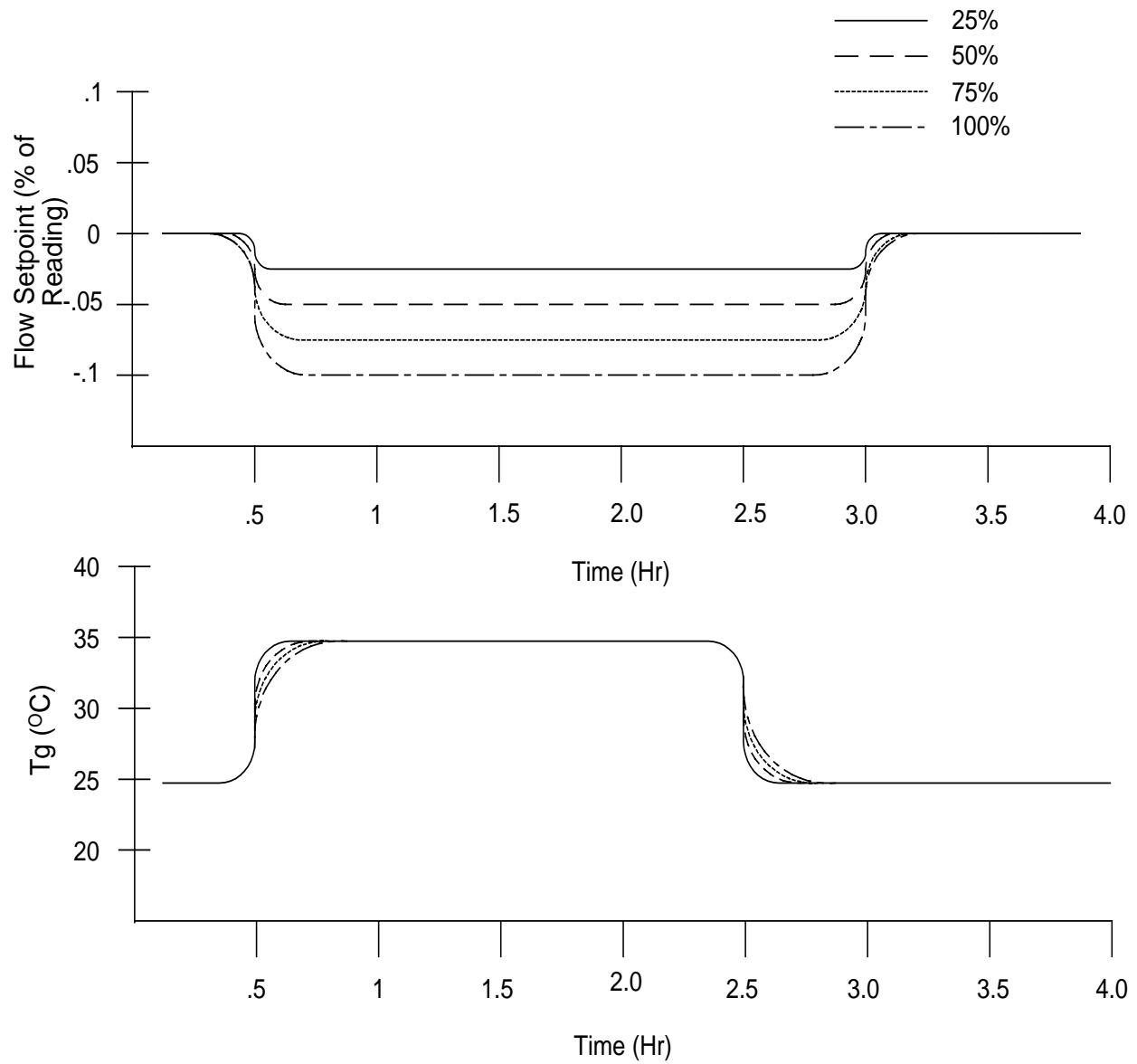
**Figure 5**  
**Steady State Ambient Temperature Effects**



**Figure 6**  
**Transient Ambient Temperature Effects**



**Figure 7**  
**Gas Temperature Effect on Steady State Span**



**Figure 8**  
**Transient Gas Temperature Effect**

**Table 1 TC<sub>e</sub> Readings**

<i>TC<sub>e</sub> (% Reading/°C)</i>								
	T <sub>e2</sub> -T <sub>e1</sub>	T <sub>e3</sub> -T <sub>e2</sub>	T <sub>e4</sub> -T <sub>e3</sub>	T <sub>e5</sub> -T <sub>e4</sub>	T <sub>e5</sub> -T <sub>e6</sub>	T <sub>e6</sub> -T <sub>e7</sub>	T <sub>e7</sub> -T <sub>e8</sub>	T <sub>e8</sub> -T <sub>e9</sub>
Flow Setpoint % F.S.								
25								
50								
75								
100								
0								

NOTE 1: Plot as in Figure 7.

**Table 2 TC<sub>g</sub> Readings**

<u>Flow Setpoint (%F.S.)</u>	<u>TC<sub>g</sub>(% reading/°C)</u>
25	-0.06
50	-0.08
75	-0.09
100	-0.11
0	

NOTE 1: Plot as in Figure 5.

**Table 3 Data Presentation for 500 sccm MFC**

<i>Data Collection</i>									
	T <sub>e1</sub> Q <sub>a1</sub>	T <sub>e2</sub> Q <sub>a2</sub>	T <sub>e3</sub> Q <sub>a3</sub>	T <sub>e4</sub> Q <sub>a4</sub>	T <sub>e5</sub> Q <sub>a5</sub>	T <sub>e6</sub> Q <sub>a6</sub>	T <sub>e7</sub> Q <sub>a7</sub>	T <sub>e8</sub> Q <sub>a8</sub>	T <sub>e9</sub> Q <sub>a9</sub>
0% S.P. Q <sub>ind</sub>									
25% S.P. Q <sub>ind</sub> T <sub>g</sub> Avg. of 10 readings									
50% S.P. Q <sub>ind</sub> T <sub>g</sub> Avg. of 10 readings									
75% S.P. Q <sub>ind</sub> T <sub>g</sub> Avg. of 10 readings									
100% S.P. Q <sub>ind</sub> T <sub>g</sub> Avg. of 10 readings									

Data Presentation

$$T_{ce} = \frac{Q_a(30) - Q_a(20)}{T_e(30) - T_e(20) Q_{s.p.}} \times 100\%$$

$$T_{ce} = \frac{134 - 122}{30.1 - 20.1 (125)} \times 100\%$$

$$= 0.96\% \text{ of Reading @ 125 sccm}$$



**Table 4 Sample Data for 500 sccm MFC**

$T_g$	$T_s$	$T_e$	$Q_{sp}$	$Q_{ind}$	$Q_A$	<i>Time min.</i>
		20.1		10		.5
		20.2		15		1
		20.3		11		1.5
		20.4		14		2
		20.5		24		2.5
		.		.		
		.		.		
		.		.		
		30.9		36		60
		.				
		.				
		.				
		40.0		48		120
		39.0		40		480
		30.0		36		560
		20.0		10		600

NOTE 1: Plot as in Figure 8.

**Table 5 Gas Temperature Data Collection**

<i>Set point (sccm)</i>	<i>T<sub>s</sub> (°C)</i>	<i>T<sub>g</sub> (°C)</i>	<i>T<sub>b</sub> (°C)</i>	<i>T<sub>e</sub> (°C)</i>	<i>Q<sub>a</sub> (sccm)</i>	<i>Q<sub>ind</sub></i>
0		22.0		21.8	0.00	
25		22.1		21.9	24.94	
25		33.2		22.2	24.52	
50		22.1		21.8	50.08	
50		32.1		21.7	49.21	
75		22.0		21.6	75.13	
75		31.8		21.5	74.02	
100		21.9		21.4	100.18	
100		31.7		21.4	98.83	

$$TC_g = \frac{Q_{af} - Q_{ai}}{[(T_g - T_e)_f - (T_g - T_e)_i] Q_{sp}} \times 100\%$$

Example: For the 50 sccm setpoint data collection sample,

$$TC_g = \frac{49.21 \text{ sccm} - 50.08 \text{ sccm}}{[(32.1^\circ\text{C} - 21.7^\circ\text{C}) - (22.1^\circ\text{C} - 21.8^\circ\text{C})](50 \text{ sccm})} \times 100\%$$

$$= -0.17\% \text{ of reading at } 50 \text{ sccm}$$

## APPENDIX 1

NOTE: This appendix is being balloted as an official part of SEMI F062-0701 by full letter ballot procedure. Determination of the suitability of the material herein is solely the responsibility of the user.

(Mandatory Information)

### A1-1 Reference Values

Ambient Temperature	23 $\pm$ 2°C
Gas Temperature	Same as Ambient
Ambient Pressure	101.3 kPa $\pm$ 4.7 kPa (1013 mbar $\pm$ 4.7 kPa - 15.3 kPa)
Gas Pressure, Inlet	274 $\pm$ 34 kPa
Gas Pressure, Outlet	101.3 kPa
Gas Pressure, Inlet	172 $\pm$ 34 kPa
Gas Pressure, Outlet	< 0.13 kPa
Relative Humidity	40% $\pm$ 10%, Noncondensing
Magnetic Field	< 50 $\mu$ T
Electromagnetic Field	< 100 $\mu$ V/m
Vibration	< 0.5 m/s
Shock	$\leq$ 3g

**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 manufacture'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 F63-0701

## GUIDELINES FOR ULTRAPURE WATER USED IN SEMICONDUCTOR PROCESSING

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 Facilities Committee on April 30, 2001. Initially available at [www.semi.org](http://www.semi.org) May 2001; to be published July 2001.

### 1 Purpose

1.1 This guide is provided for multiple purposes. It may be used as a basis for establishing performance criteria for purchases of new UPW equipment. It may also be used internally by facility engineers to set process control parameters for the operation of their UPW systems. This guide may be used by process engineers to establish reasonable expectations about the quality of the UPW being supplied to them by facilities.

NOTICE: These suggested guidelines are published as technical information and are intended for informational purposes only.

### 2 Scope

2.1 Water is used extensively in the production of semiconductor devices for all wet processing steps such as the rinsing of wafers. Ultrapure Water (UPW) is typically produced for this purpose using Reverse Osmosis/Deionized resin bed technologies. The quality of the water impacts device yield and as linewidths decrease, requirements for higher purity water may increase.

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

### 3 Referenced Standards

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

#### 3.1 SEMI Standards

SEMI F61 — Guide For Ultrapure Water System Used in Semiconductor Processing

#### 3.2 ASTM Standards<sup>1</sup>

ASTM D-5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry.

ASTM F-1094 — Test Method For Microbiological Monitoring of Water Used For Processing Electron and Microelectronic Devices by Direct- Pressure Tap Sampling Valve and by the Pre-sterilized Plastic Bag Method.

### 4 Limitations

4.1 This guide is not intended to set an absolute number for performance of a particular UPW system. Performance guidelines should be determined based on the design of the UPW system, the components used in the water system, the sensitivity of the manufacturing process to the purity of the water, the sensitivity of the instrumentation, and the budget available to maintain and monitor the water system.

4.2 This set of guidelines has been established from a variety of sources and inputs including 1.) an industry survey that SEMI provided to members of its standards activities in facilities; 2.) typical UPW levels from a large selection of semiconductor UPW systems as measured by several independent laboratories that test high purity water for the semiconductor industry; 3.) specifications from water system equipment manufacturers; 4.) and input from producers and users of UPW during SEMI standards committee meetings and through the balloting process. However, it is up to each individual owner of a UPW system to set specifications for the purity of its water based on its own needs and available resources.

4.3 This guide is reflective of a particular design approach for a UPW system. The guidelines can be produced from a properly maintained UPW system as described and diagrammed in SEMI F61 and is typical output from existing high-end semiconductor manufacturing plants with state-of-the-art water systems. The purity of water generated in other types of water systems may differ greatly.

4.4 The guidelines are targeted to UPW produced for semiconductor manufacturing. There exists an ASTM standard D5127 which should be considered in setting specifications for the purity of reagent grade water needed in a laboratory for analytical testing of semiconductor materials and process chemicals.

<sup>1</sup> American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, website: [www.astm.org](http://www.astm.org)

## 5 Use of the Guidelines

5.1 Sampling methods and contamination control are of paramount importance when attempting to measure the listed parameters at the levels specified.

5.2 The quality of the data measured will depend on which testing method and calibration techniques are used. Consequently, trends observed in the values may be more meaningful than absolute values.

## 6 Units

6.1 Parts per billion (ppb) is equivalent to ng/mL or µg/L.

6.2 Parts per million (ppm) is equivalent to mg/L.

6.3 Micron is a unit of length equal to one millionth of a meter, or one thousandth of a millimeter.

6.4 Colony Forming Units (CFU) is a measurement of bacteria organisms.

## 7 Description of Parameter Tests

NOTE 2: Since SEMI Guidelines do not require analytical data or methods to support them, the recommendation of specific analytical methods are only for informational purposes. Alternative methods may also be applicable.

### 7.1 Resistivity (megohm-centimeters or Mohm-cm)

7.1.1 Resistivity (conductivity) is only measured accurately with on-line instrumentation. 18.25 MOhm is the theoretical upper limit for pure water at 25°C.

### 7.2 Total Oxidizable Carbon (TOC) (ppb)

7.2.1 Involves oxidation of organic materials and detection of carbon dioxide produced by the reaction, as measured in conductivity or infrared photometry.

7.3 Dissolved oxygen (ppb) is only measured accurately with on-line instrumentation.

### 7.4 Particulate Matter (Particles/L)

7.4.1 On-line methods using laser technology are recommended for accurate trend analysis.

### 7.5 Bacteria (CFU/L)

7.5.1 Triplicate samples are cultured based on the ASTM method F 1094 using a minimum sample size of 1L.

### 7.6 Silica

7.6.1 Total Silica (ppb) may be measured by Graphite Furnace Atomic Absorption Spectrophotometry (GFAAS), Inductively-Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) or Inductively Coupled Plasma Mass Spectroscopy (ICP-MS).

7.6.2 Dissolved Silica (ppb as SiO<sub>2</sub>) may be measured by heteropoly blue photometry or by Ion Chromatography.

### 7.7 Ions and Metals (ppb)

7.7.1 Many anions and cations may be determined using Ion Chromatography.

7.7.2 Up to 68 metals may be determined by GFAAS, ICP-AES, or ICP-MS. Most typically measured metals are shown in Table 1.

## 8 Parameters and Typical Concentrations

8.1 Table 1 lists each parameter with its range of performance.

**Table 1 Parameters and Range of Performance**

<i>Typical Linewidth</i>	<i>0.13 to 0.5 MICRONS</i>
<b>PARAMETER</b>	<b>RANGE OF PERFORMANCE</b>
<b>Resistivity on-line @ 25°C (Mohm-cm)</b>	17.9 to 18.2
<b>TOC on-line (ppb)</b>	1 to 5
<b>Dissolved Oxygen on-line (ppb)</b>	0.5 to 20
<b>On-line Particles/L (micron range)</b>	
0.05-0.1	100 to 1000
0.1-0.2	50 to 500
0.2-0.3	20 to 100
0.3-0.5	10 to 50
> 0.5	0 to 4
<b>Bacteria (CFU/L)</b>	
1 L Sample	0 to 5
<b>Silica</b>	
Silica - total (ppb)	0.5 to 3
Silica - dissolved (ppb as SiO <sub>2</sub> )	0.2 to 1
<b>Ions &amp; Metals (ppb)</b>	
Ammonium	0.02 to 0.1
Bromide	0.02 to 0.1
Chloride	0.02 to 0.1
Fluoride	0.02 to 0.1
Nitrate	0.02 to 0.1
Nitrite	0.02 to 0.1
Phosphate	0.02 to 0.1
Sulphate	0.02 to 0.1
Aluminum	0.02 to 0.1
Barium	0.02 to 0.1
Boron	0.02 to 20
Calcium	0.02 to 0.1
Chromium	0.02 to 0.1

<i>Typical Linewidth</i>	<i>0.13 to 0.5 MICRONS</i>
Copper	0.02 to 0.1
Iron	0.02 to 0.1
Lead	0.02 to 0.1
Lithium	0.02 to 0.1
Magnesium	0.02 to 0.1
Manganese	0.02 to 0.1
Nickel	0.02 to 0.1
Potassium	0.02 to 0.1
Sodium	0.02 to 0.1
Strontium	0.02 to 0.1
Zinc	0.02 to 0.1

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# SEMI F64-0701

## TEST METHOD FOR DETERMINING PRESSURE EFFECTS ON INDICATED AND ACTUAL FLOW FOR MASS FLOW CONTROLLERS

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 Facilities Committee on April 30, 2001. Initially available at [www.semi.org](http://www.semi.org) May 2001; to be published July 2001.

### 1 Purpose

1.1 The purpose of this document is to define a method for characterizing a MFC being considered for installation into a gas distribution system. This method will quantify the effect of transient and steady state inlet and outlet pressure conditions on the performance of the MFC.

1.2 This document provides a common basis for communication between manufacturers and users regarding testing and describing MFC pressure effects.

### 2 Scope

2.1 This test method measures the upstream (inlet) and downstream (outlet) transient pressure influences on indicated and actual flow.

2.2 This test method yields the results of actual output flow versus MFC set-point and indicated flow as influenced by steady state inlet pressure.

2.3 This test method applies to MFCs with maximum flow ranges of up to 1000 sccm.

NOTE 1: Due to the higher sensitivity of lower flow rate MFC's when pressure transients occur, the flow range for this document is limited to 1000 sccm.

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

### 3 Limitations

3.1 This test method is limited to analyzing the effect of pressure on performance characteristics of MFCs and is not a verification of the state of calibration, linearity, or accuracy.

3.2 This test method does not address pressures in excess of the DUT's maximum working pressure as specified by the manufacturer.

3.3 This test method is limited to reasonable pressure transients; i.e., fluctuations that are otherwise tolerated by common semiconductor process equipment.

3.4 This test method does not address operational influences outside of the manufacturer's published limitations.

3.5 This test method does not address the effects of interruptions of the gas supply.

### 4 Referenced Standards

4.1 None.

### 5 Terminology

#### 5.1 Definitions

5.1.1 *actual flow* — flow as indicated by flow standard (see Figures 1 and 2).

5.1.2 *indicated flow* — flow as indicated by the device under test (DUT).

5.1.3 *ramp* — constant rate of change in pressure ( $dp/dt = k$ ).

5.1.4 *stability* — the ability of a condition to exhibit only natural, random variation in absence of unnatural, assignable cause variation.

5.1.5 *step change* — an exponential step in pressure with a time constant of one second or less.

#### 5.2 Abbreviations and Acronyms

5.2.1  $\delta M$  — deviation of mass of material relative to steady-state mass delivery.

5.2.2  $\Delta P$  — change in pressure with respect to time

5.2.3  $\Delta Q$  — steady state deviation of actual flow during inlet pressure ramp from that while inlet pressure is constant.

5.2.4  $\delta Q_-$  — maximum negative deviation of actual flow from nominal.

5.2.5  $\delta Q_+$  — maximum positive deviation of actual flow from nominal.

5.2.6  $\tau$  — pneumatic time constant

5.2.7 *DUT* — device under test

5.2.8 *MFC* — mass flow controller

5.2.9 *MV* — metering valve

- 5.2.10  $P_I$  — inlet pressure
- 5.2.11  $P_2$  — outlet pressure
- 5.2.12  $PC_A$  — pressure coefficient of actual flow per pressure change at a set point.
- 5.2.13  $PC_O$  — pressure coefficient of indicated flow per pressure change at zero flow.
- 5.2.14  $PC_S$  — pressure coefficient of span flow per pressure change.
- 5.2.15  $Psia$  — pounds per square inch absolute
- 5.2.16  $Psig$  — pounds per square inch gauge
- 5.2.17  $Q_A$  — actual flow
- 5.2.18  $Q_{FS}$  — rated full scale flow
- 5.2.19  $Q_I$  — indicated flow
- 5.2.20  $Q_N$  — nominal actual flow during steady state conditions.
- 5.2.21  $Q_R$  — steady state actual flow while inlet pressure is being ramped.
- 5.2.22  $Q_{SP}$  — set-point flow
- 5.2.23  $T$  — time
- 5.2.24  $t_f$  — time when  $Q_A$  is within 0.5% of reading of  $Q_N$ .
- 5.2.25  $t_o$  — time when pressure transient is initiated.
- 5.2.26  $t_s$  — settling time to  $Q_N$
- 5.2.27  $v$  — voltage
- 5.2.28  $V_{eq}$  — equivalent internal control volume of the DUT.
- 5.2.29  $V_{ip}$  — valve, pump isolation

## 6 Summary of Test Method

6.1 *Inlet Pressure Step and Ramp Change* — The effects of fast-step and slow-ramp changes to the pressure on actual flow out of the MFC is observed. See Figures 6 and 7.

6.2 *Inlet Pressure Effect Steady State* — The effects of a pressure increase/decrease on actual flow is observed once the increase/decrease has reached steady state. See Figure 8.

6.3 *Outlet Pressure Step Change* — The effects of an outlet pressure change on actual flow is observed. See Figure 9.

6.4 *Crosstalk Pressure Effect* — The effects of pressure changes due to switching multiple flow devices on the same gas line. See Figure 11.

## 7 Interferences

7.1 The accuracy rating of the measuring equipment shall be superior to that of the DUT. Preferably the measuring equipment will have an accuracy that is four times better than the DUT. Calibration equipment must have a valid calibration certificate.

7.2 Take care when using test instruments with a specified accuracy expressed in percent of full scale as the accuracy is limited at lower percentages.

7.3 Installation effects on the flow should be minimized.

7.4 Verify electrical signals directly at the DUT connector to ensure that the signals at the DUT and standard agree with the signals at the data recording equipment.

7.5 All electrical measurements should be read on devices with at least 4.5 digits of resolution. These devices must have valid calibration certifications.

7.6 The results of this test method depend on the accuracy and repeatability of the pressure measurement devices used in the test system. Take care to stay within the specified pressure ranges of these devices and verify their accuracy prior to and following MFC evaluation activities.

7.7 The cleanliness level of the test gas should be compatible with the DUT. The minimum requirement placed on the test gas is that it be free of contamination that could influence the operational characteristics of the test article and instruments.

7.8 The test gas source and delivery system must be capable of satisfying the test volume flow rate at a constant pressure  $\pm 0.7$  kPa ( $\pm 0.1$  psi).

7.9 The ambient temperature should be held to 22°C  $\pm 2^\circ\text{C}$  for the duration of each analysis.

## 8 Significance and Use

8.1 The significance of the stability calculations in this method is to allow the MFC user to assess the transient pressure effects on the DUT. In application, this method will provide a consideration affecting gas system designs and MFC selection.

## 9 Apparatus

### 9.1 Equipment Required for Methods A-1 and A-2

9.1.1 Inlet test gas filter

9.1.2 Shutoff valve (qty 2).

9.1.3 Pressure transducers, available range 0–446 kPa (0–50 psig), with measurement accuracy of  $\pm 0.2\%$  and time constant less than 20 msec (qty 2).



9.1.4 Dome-loaded pressure control, or other device capable of producing specified pressure transients.

9.1.5 Flow standard, with time constant less than 20 msec and with full scale that is 200% of the DUT's full scale, accurate to 1% of full scale, linear to 0.5% of full scale, and capable of resolving to 0.2% of full scale.

9.1.6 Data acquisition system measures the electrical signals from the device under test. The data acquisition system may also read the signals from the flow standard, record test data and control the test sequence.

## 9.2 *Equipment Required for Method B*

9.2.1 Inlet test gas filter.

9.2.2 Shutoff valve (qty 2).

9.2.3 Pressure transducer, available range 0–791 kPa (0–100 psig), with measurement accuracy of  $\pm 0.1\%$  and time constant less than 20 msec.

9.2.4 Two-stage pressure regulator, capable of 0–690 kPa (100 psia) control.

9.2.5 Flow standard, with full scale that is at least 120% of the DUT's full scale, accurate to 1% of full scale, linear to 0.5% of full scale, and capable of resolving to 0.2% of full scale.

9.2.6 Data acquisition system measures the electrical signals from the device under test. The data acquisition system may also read the signals from the flow standard, record test data and control the test sequence.

## 9.3 *Equipment Required for Method C*

9.3.1 Inlet test gas filter.

9.3.2 Manual metering valve.

9.3.3 Pressure transducer, available range 0–446 kPa (0–50 psig), with measurement accuracy of  $\pm 0.2\%$  and time constant less than 20 msec.

9.3.4 Pressure transducer, 0–101.325 kPa  $\pm 1\%$  and with time constant less than 20 msec (qty 1).

9.3.5 Shutoff valves (qty 2).

9.3.6 Isolation valve (qty 1).

9.3.7 Vacuum pump, with pumping speed of at least 30 lpm, with throttling and isolation valve.

9.3.8 Flow standard, with time constant less than 20 msec and with full scale that is 200% of DUT full scale accurate to 1% of full scale, linear to 0.5% of full scale, and capable of resolving to 0.2% of full scale.

9.3.9 Data acquisition system measures the electrical signals from the device under test. The data acquisition system may also read the signals from the flow standard, record test data and control the test sequence.

## 9.4 *Equipment Required for method D*

9.4.1 Pressure transducer, available range 0–446 kPa (0–50 psig), with measurement accuracy of  $\pm 0.2\%$  and time constant less than 20 msec. (2)

9.4.2 Flow standard, with time constant less than 20 msec and with full scale that is 200% of DUT full scale accurate to 1% of full scale, linear to 0.5% of full scale, and capable of resolving to 0.2% of full scale.

9.4.3 Isolation valve (qty 1).

9.4.4 Data acquisition system measures the electrical signals from the device under test. The data acquisition system may also read the signals from the flow standard, record test data and control the test sequence.

9.4.5 MFC with time constant less than 20 msec and a full scale flow rate that is 10 times the full scale flow rate of the DUT.

## 10 **Materials**

10.1 Clean, dry nitrogen, with a dew point less than or equal to  $-40^{\circ}\text{C}$ , at a delivery pressure of 791 kPa (100 psig).

## 11 **Safety Precautions**

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

## 12 **Test Specimen**

12.1 Allow all components in the test apparatus to warm up following the manufacturer's specification.

12.2 Take necessary steps when switching gases to ensure that only the desired gas is in the DUT and flow standard at the time the test is performed.

## 13 **Preparation of Apparatus**

13.1 *Setup and Schematic* — See Figures 1, 2, and 10.

13.2 *Plumbing Design Requirements for Methods A and C*

13.3 The test system must be highly conductive and must have no changes in direction closer than 20 tubing diameters upstream of the DUT.

13.4 The pneumatic time constant for the system shall be calculated as shown below:

$$\tau = (\text{Volume} \times \text{Pressure Drop}) / (\text{Pressure} \times \text{Volumetric Flow})$$

where:

- Volume is measured between the flow standard and the DUT
- Pressure drop is across the flow standard
- Pressure is the pressure in the defined volume
- Volumetric flow is as measured by the standard

13.5 The pneumatic time constant added to the flow standard time constant should ideally be less than 100 msec and should be recorded if larger. For method C, the volume between MV and  $V_{ip}$  should be kept to a minimum.

13.6 The system must be relatively leak free, i.e., the maximum inboard leak rate should be  $2 \times 10^{-7}$  atm cc/s He.

13.7 The system must be supported and isolated in a manner that keeps it from vibration, shock, and other conditions that could influence the results of this test.

## 14 Procedure

### 14.1 Inlet Transient Pressure Effects (Method A-1)

14.1.1 Assemble the test MFC into the test apparatus. (See Figure 1.)

14.1.2 Satisfy the manufacturer's warm-up requirements for the MFC.

14.1.3 Set the inlet pressure ( $P_1$ ) to 274 kPa (25 psig).

14.1.4 Give the DUT a 50% set-point ( $Q_{SP} = \frac{1}{2}Q_{FS}$ ) and wait for  $Q_I$  to reach stability.

14.1.5 Adjust the pressure control device to initiate a step in  $P_1$  to 287 kPa (27 psig) with a time constant of one second (see Figure 3c).

14.1.6 Wait until  $Q_I$  and  $Q_A$  have reached stability.

14.1.7 Using the pressure control device, initiate a step in  $P_1$  to 274 kPa (25 psig) with a time constant of one second.

14.1.8 Give the DUT a 100% set-point ( $Q_{SP} = Q_{FS}$ ) and wait for stability.

14.1.9 Repeat Sections 14.1.5 through 14.1.7.

### 14.2 Process Side Effects (Method A-2)

14.2.1 Assemble the test MFC into the test apparatus (see Figure 1).

14.2.2 Satisfy the manufacturer's warm-up requirements for the MFC.

14.2.3 Set the inlet pressure ( $P_1$ ) to 274 kPa (25 psig).

14.2.4 Give the DUT a 50% set point ( $Q_{SP} = \frac{1}{2}Q_{FS}$ ) and wait for  $Q_I$  to reach stability.

14.2.5 Using the pressure control device, initiate a ramp in  $P_1$  to 356 kPa (37 psig) (see graph in Figure 4c). Use a ramp rate between 0.7 kPa (0.1 psi) per second and 21.0 kPa (3psi) per second. Record the ramp rate used on the data sheet.

14.2.6 Wait until  $Q_I$  and  $Q_A$  have reached stability.

14.2.7 Using the pressure control device, initiate a ramp as in 14.2.5, initiate a ramp in  $P_1$  to 274 kPa (25 psig) (see graph in Figure 4c).

14.2.8 Repeat Sections 14.2.5 through 14.2.7.

### 14.3 Inlet Pressure Effects, Steady State (Method B)

14.3.1 Assemble the DUT in the test apparatus (see Figure 1).

14.3.2 Satisfy the manufacturer's warm-up requirements for the DUT.

14.3.3 Set the inlet pressure ( $P_1$ ) to 205 kPa (15 psig).

14.3.4 Close  $V_{io}$ , open DUT control valve.

14.3.5 Wait until  $Q_R$  and  $Q_A$  have reached stability. Record  $P_1$ ,  $Q_I$  and  $Q_A$  (see Table A3.1).

14.3.6 Open  $V_{io}$  and give the DUT a 50% set-point. ( $Q_{SP} = \frac{1}{2}Q_{FS}$ ).

14.3.7 Wait until  $Q_I$  and  $Q_A$  have reached stability. Record  $P_1$ ,  $Q_I$  and  $Q_A$  (see Table A3.1).

14.3.8 Give the DUT a 100% set-point ( $Q_{SP} = Q_{FS}$ ).

14.3.9 Wait until  $Q_I$  and  $Q_A$  have reached stability. Record  $P_1$ ,  $Q_I$  and  $Q_A$  (see Table A3.1).

14.3.10 Set inlet pressure ( $P_1$ ) to 446 kPa (50 psig) and repeat Sections 14.3.4 through 14.3.9.

### 14.4 Outlet Pressure, Step Change (Method C)

14.4.1 Assemble the test MFC into the test apparatus. (See Figure 1.)

14.4.2 Satisfy the manufacturer's warm-up requirements for the MFC.

14.4.3 Set the inlet pressure ( $P_1$ ) to 274 kPa (25 psig) (see Figure 9).

14.4.4 Give the DUT a 50% set point ( $Q_{SP} = \frac{1}{2}Q_{FS}$ ).

14.4.5 Throttle the pump rate until  $P_2 = 26.664$  kPa.

14.4.6 Open  $V_{io}$  and adjust the metering valve, MV, until  $P_2 = 101.325$  kPa.

14.4.7 Allow  $Q_I$  to reach stability. Record  $P_1$ ,  $Q_I$ , and  $Q_A$  (see Table A3).

14.4.8 Close  $V_{io}$ .  $P_2$  should = 26.664 kPa.

14.4.9 Allow  $Q_I$  to reach stability. Record  $P_1$ ,  $Q_I$ , and  $Q_A$  (see Table A3).

14.4.10 Give the DUT a 100% set point and repeat Sections 14.4.5 through 14.4.9.

#### 14.5 Crosstalk Pressure Effect (Method D)

14.5.1 Assemble the DUT in the test apparatus (see Figure 10).

14.5.2 Satisfy the manufacturer's warm-up requirements for the DUT.

14.5.3 Set the inlet pressure ( $P_1$ ) to 274 kPa (25 psig)

14.5.4 Give the DUT a 100% set point ( $Q_{SP} = Q_{FS}$ ).

14.5.5 Give the 10× MFC a 100% set point ( $Q_{SP} = Q_{FS}$ ).

14.5.6 Wait until the DUT and MFC achieve stability.

14.5.7 Record  $P_1$ ,  $P_2$ , the indicated flow from the DUT, the indicated flow from the MFC and the indicated flow from the flow standard continuously with the data acquisition system.

14.5.8 While monitoring command the 10 × MFC to a zero set-point. Wait until the DUT becomes stable then command the 10× MFC to a 100% set Point.

14.5.9 Sample Plot is shown in Figure 11.

### 15 Calculations or Interpretation of Results

15.1 See Appendix 1 for detailed information on data collection and interpretation for Methods A-1, A-2, B, and C.

15.2 *Calculations* — The indicated MFC flow output data and the flow standard output data should be converted to percent of full scale flow as follows:

$$\% \text{ of full scale flow} = \frac{\text{output data (v)}}{\text{full scale output (v)}} \times 100 \quad (1)$$

15.3 *Interpretation* — Collect the following data and report as an effect of transient pressure conditions:

15.4 Plot of indicated flow, actual flow, and flow set point versus time.

15.5 Duration of instability. Maximum and minimum deviations from the steady state actual and indicated flows. Calculate the positive and negative integrals, along with the total integral of variations in the actual and indicated flows.

15.6 Inlet ( $P_1$ ) and outlet ( $P_2$ ) values.

15.7 The number of repetitions required to achieve a confidence factor of 95% for each test step.

15.8 Data plots for the measured values must be generated and presented in a manner similar to that shown in Figures 3, 4, and 5 and in Table 1.

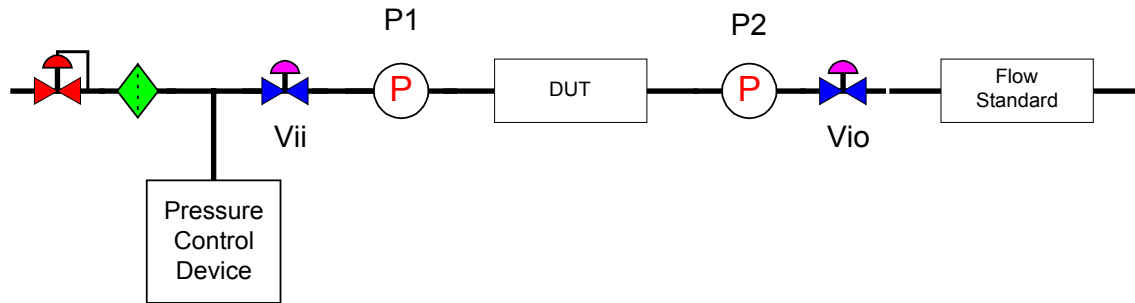
### 16 Related Documents

16.1 *Military Standards*<sup>1</sup>

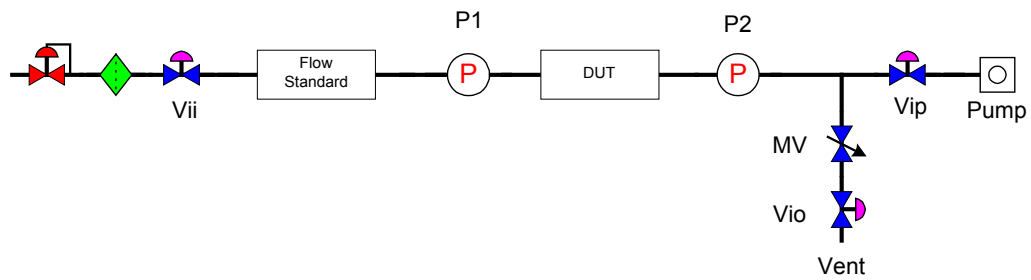
MIL-STD 45662 — Calibration Systems Requirements

<sup>1</sup> Naval Publication and Forms Center, 5801 Tabor Avenue, Philadelphia PA 19120 U.S.A

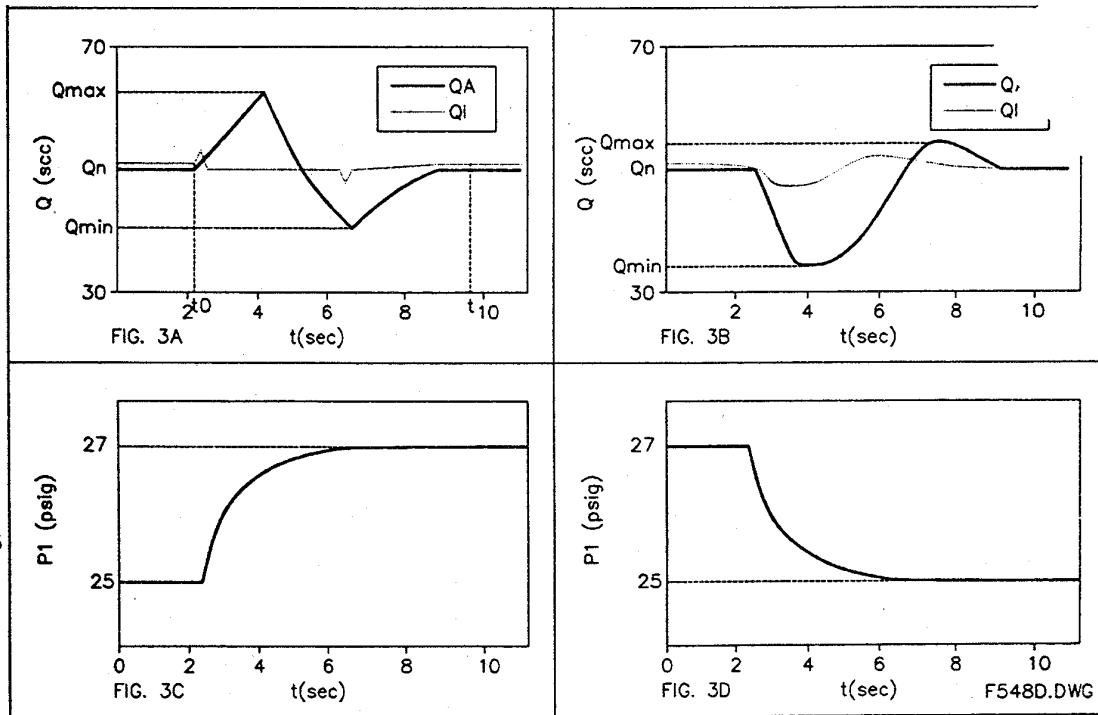
## 17 Illustrations



**Figure 1**  
**Test Schematic for Methods A, A<sub>1</sub>, A<sub>2</sub>, and B**



**Figure 2**  
**Test Schematic for Method C**



### Figure 3

### Figure 4

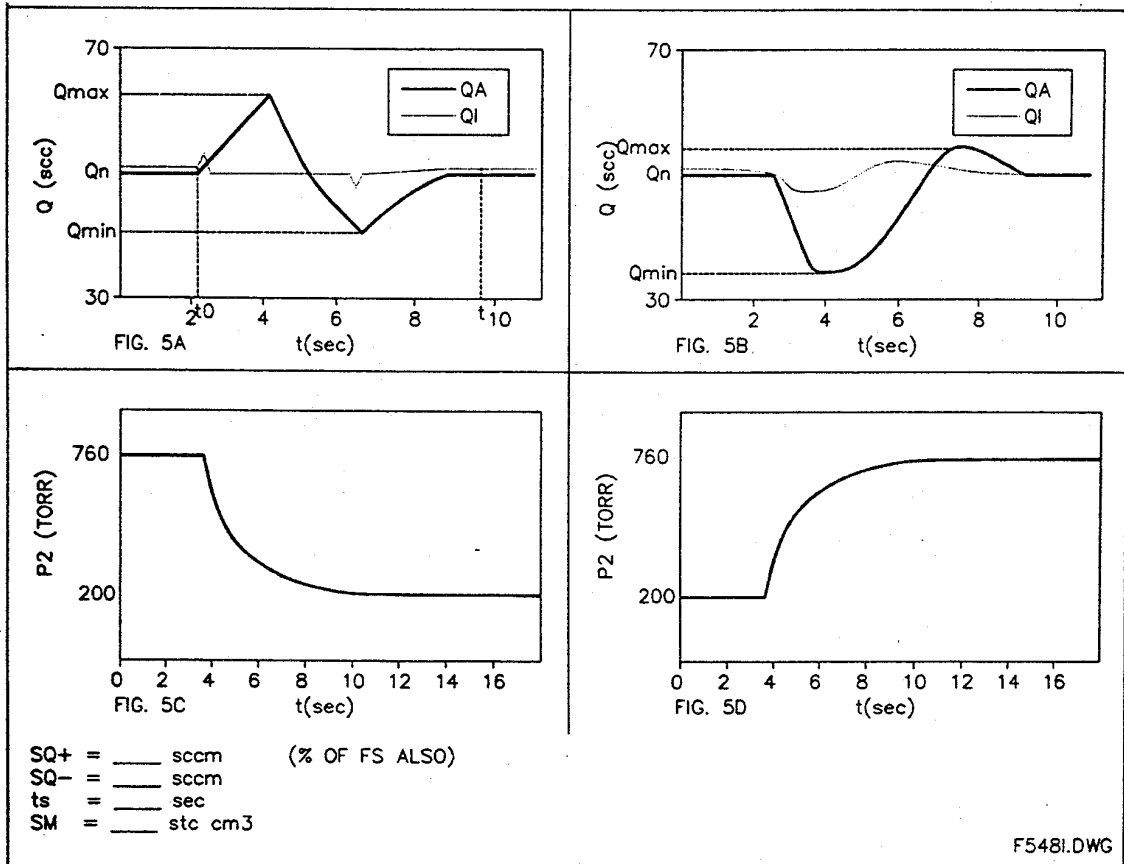
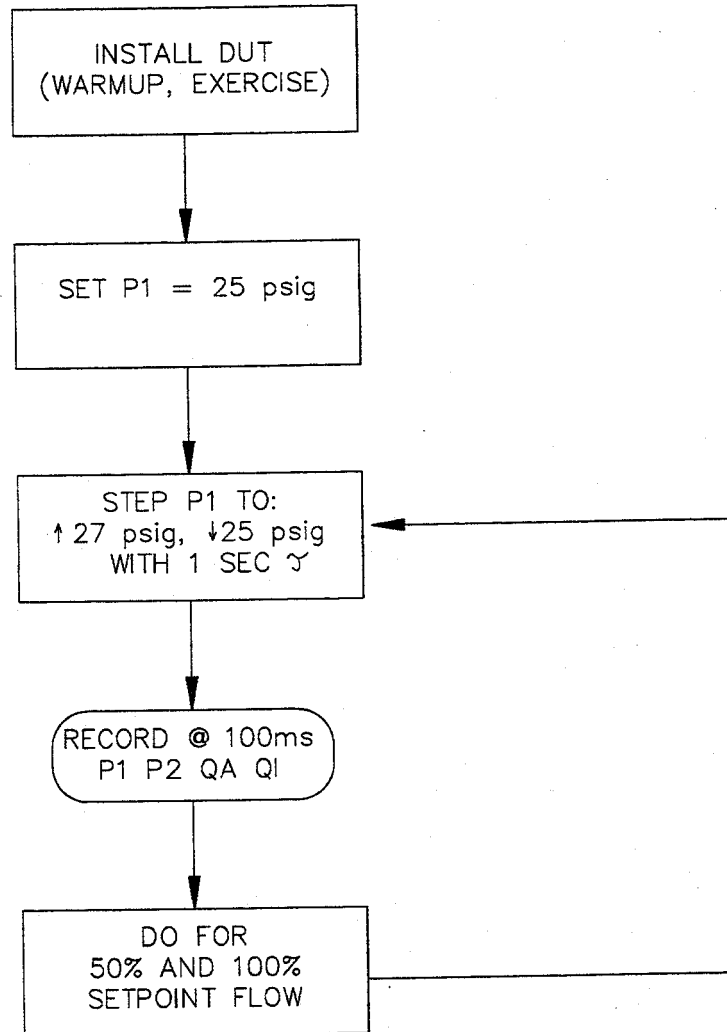
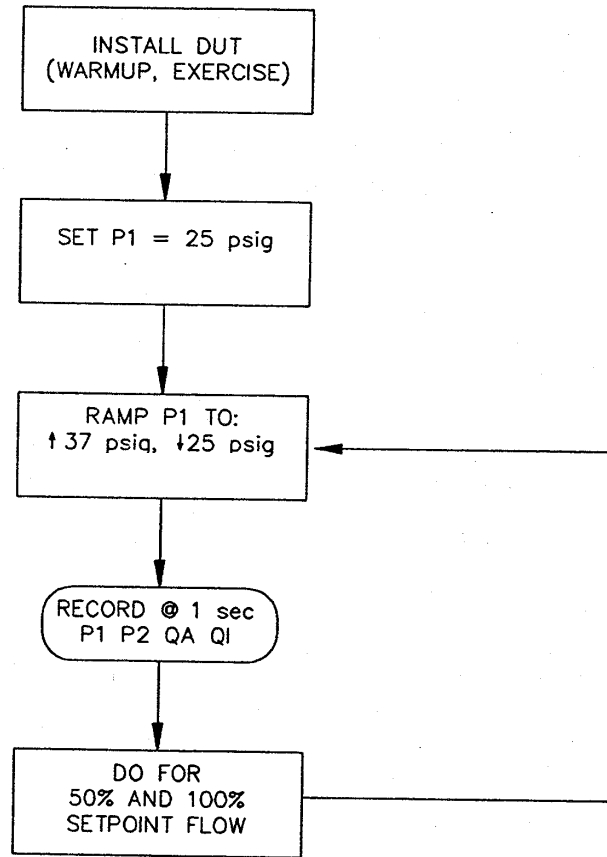


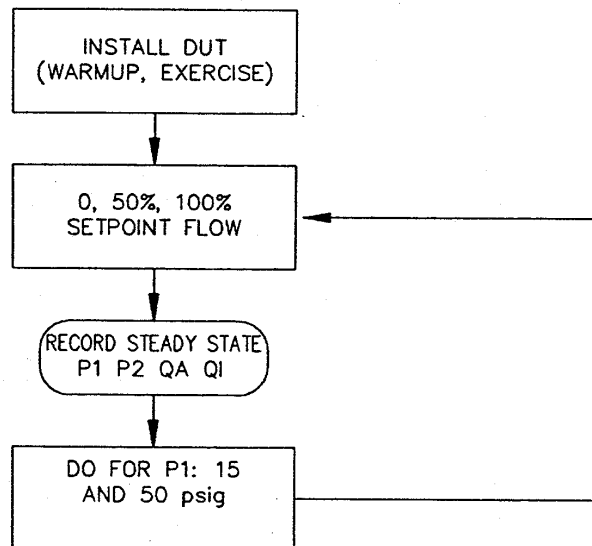
Figure 5  
Q and P<sub>2</sub> versus t: Data Presentation for Method C (see Appendix 1)



**Figure 6**  
**Method A-1 Flowchart**

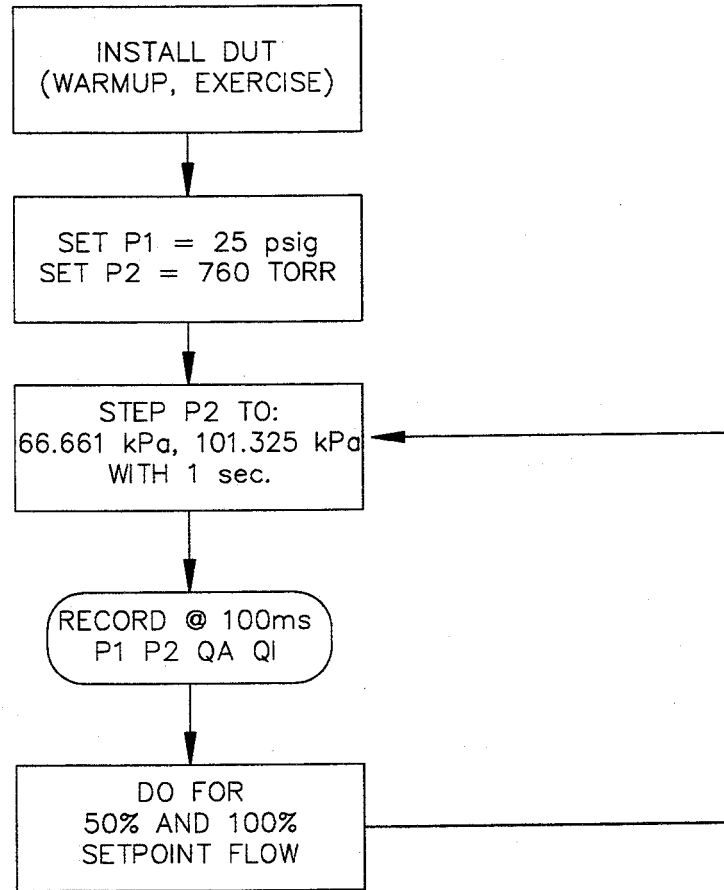


**Figure 7**  
**Method A-2 Flowchart**

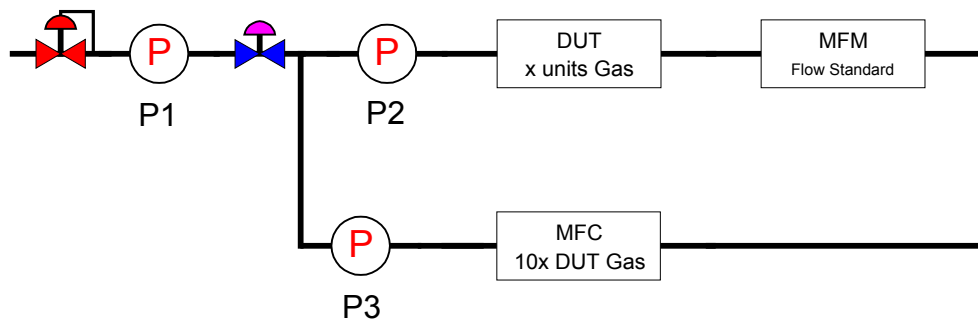


**Figure 8**  
**Method B Flowchart**

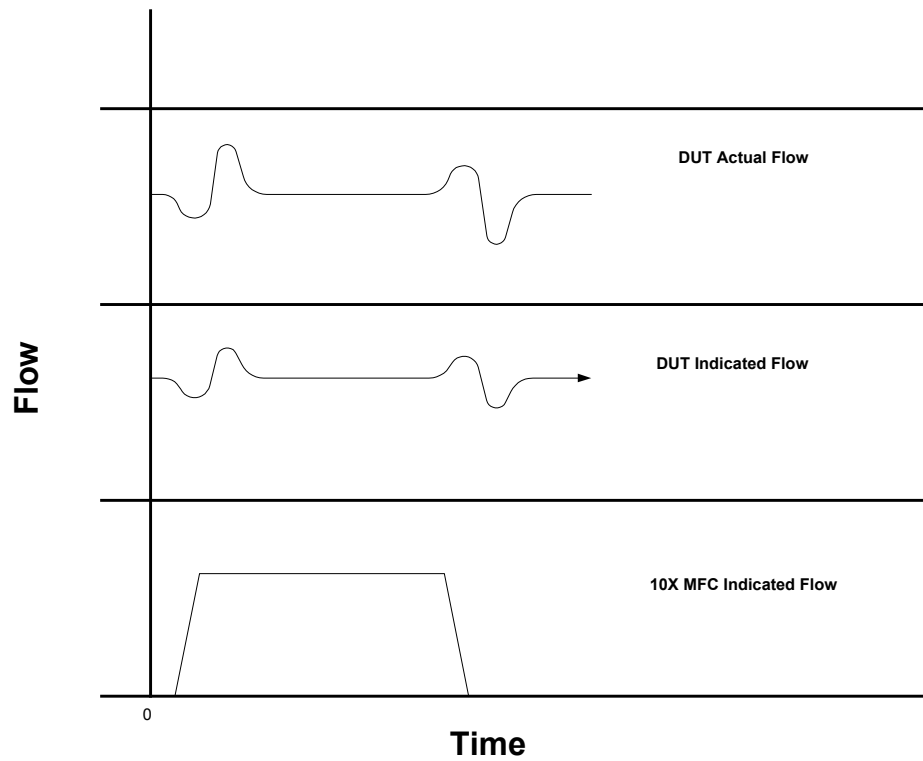




**Figure 9**  
**Method C Flowchart**



**Figure 10**  
**Test Schematic for Method D**



**Figure 11 Plots  
Test Method D**

**Table 1 Data Presentation for Method B (see Appendix 1)**

**PC<sub>0</sub> = \_\_\_\_\_ % of FS/psi**

	@ 50% FS	@ FS
PC <sub>A</sub> (% of reading/psi)		