SEMASPEC Guide to Provisional Test Methods for Mass Flow Controllers

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Technology Transfer 92071220B-STD

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Abstract: This guide provides supplementing information about several test procedures available for testing mass flow controllers (MFCs). It is also intended to serve as a summary of the methods. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. It does not provide detailed information sufficient for conducting the procedures, but rather discusses and cites the applicable test method or standard. This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

Keywords: Testing, Mass Flow Controllers, Gas Distribution Systems

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1 Introduction

1.1 *Purpose*—This guide provides supplementary information about several test procedures available for testing mass flow controllers (MFCs). It is also intended to serve as a summary of the methods.

1.2 *Scope*—This guide provides a brief overview of applicable procedures for evaluating the performance characteristics of MFCs.

[Note: The test methods can be used sequentially or individually. However, initial tests on a MFC prior to doing a following sequence of tests can invalidate the specimen and data resulting from the sequence of tests. Therefore, the sequence of tests should be carefully planned with the understanding that several specimens are required to complete the battery of all applicable component tests without invalidating later tests. This requirement of several specimens is in addition to the replicates normally required under good statistical practices.

Because some of these tests are destructive to or can contaminate an MFC, a single MFC cannot provide valid results for a test sequence. For example, to perform a selected series of 14 test methods on a particular manufacturer's MFC and obtain valid data, several representative MFCs are required. Eight MFCs may give one data reading for each test. If the user wishes to have three readings per test, 24 MFCs are required for each test.

The MFC test methods are grouped in this guide using a sequence in which these tests are best performed so that the maximum use can be made from the minimum number of MFCs.]

1.3 Limitations

1.3.0 This guide does not provide detailed information sufficient for conducting the procedures. It is the responsibility of the user to procure a copy of the detailed test procedure from the issuing organization.

1.3.1 Test methods referenced from standards organizations (e.g., ASTM, SEMI, or EIA) are under revision control by those organizations. SEMATECH does not warrant or endorse these documents. SEMATECH is not responsible for the application and use of the methods listed in this guide or for any liabilities incurred therein.

2 Referenced Documents

2.1 ANSI[1]

ANSI B46	Surface Texture (Surface Roughness, Waviness, and Lay)

2.2 ASTM[2]

ASTM E45	Practice for Determining the Inclusion Contents of Steel

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

[2] American Society for Testing and Materials. 1916 Race St., Philadelphia, PA 19103.

2.3	EIA[3]	
	EIA RS-414-A	Simulated Shipping Tests for Consumer Electronic Products and Electronic Components
2.4		FED-STD-209[4] Federal Standard Clean Room and Work Station Requirements, Controlled Environment.
2.5	Military Standards[5]	
	MIL-STD-461C	Electromagnetic Emission and Susceptibility Requirements for the Control of Electromagnetic Interference, June 1986.
	MIL-STD-462	Measurement of Electromagnetic Interference Characteristics, July 1967.
2.6	SAMA[6]	
	SAMA PMC-33.1	Electronic Susceptibility of Process Control Instrumentation, Scientific Apparatus Makers Association, 1978.
2.7	SEMATECH[7]	
	SEMASPEC 92071221B-STD	SEMATECH Provisional Test Method for Determining Accuracy, Linearity, Repeatability, Short Term Reproducibility, Hysteresis, and Deadband of Thermal Mass Flow Controllers
	SEMASPEC 92071222B-STD	SEMATECH Provisional Test Method for Determining Reproducibility and Zero Drift for Thermal Mass Flow Controllers
	SEMASPEC 90120391B-STD	SEMATECH Test Method for the Determination of the Helium Leak Rate for Gas Distribution System Components
	SEMASPEC 92071223B-STD	SEMATECH Provisional Test Method for Determining Warmup Time of Mass Flow Controllers

[3]Electronic Industries Association. 2001 Pennsylvania Avenue, NW 9th Floor. Washington, D.C. 20006

[4]Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philidelphia, PA 19120

[5]Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, PA 19120

[6]Process Measurements & Control Section, SAMA, 1101 16th St., N. W. Washington, DC 20036

[7]SEMATECH. 2706 Montopolis Dr. Austin, TX 78741.

SEMASPEC 92071224B-STD	SEMATECH Provisional Test Method for Determining Reliability of a Mass Flow Controller
SEMASPEC 92071225B-STD	SEMATECH Provisional Test Method for Verification of Calibration Accuracy and Calculation of Conversion Factors for a Mass Flow Controller Using Surrogate Gases
SEMASPEC 92071226B-STD	SEMATECH Provisional Test Method for Determining Particle Contribution by Mass Flow Controllers
SEMASPEC 92071227B-STD	SEMATECH Provisional Test Method for Determining Moisture, Oxygen, and Total Hydrocarbon Contribution/Retention by Mass Flow Controllers
SEMASPEC 90120400B-STD	SEMATECH Test Method for the Determination of Surface Roughness by Contact Profilometry for Gas Distribution System Components
SEMASPEC 90120401B-STD	SEMATECH Test Method for SEM Analysis of Metallic Surface Condition for Gas Distribution System Components
SEMASPEC 90120402B-STD	SEMATECH Test Method for EDX Analysis of Metallic Surface Condition for Gas Distribution System Components
SEMASPEC 90120403B-STD	SEMATECH Test Method for ESCA Analysis of Surface Composition and Chemistry of Electropolished Stainless Steel Tubing for Gas Distribution System Components
SEMASPEC 90120404B-STD	SEMATECH Test Method for the Determination of Surface Roughness by Scanning Tunneling Microscopy for Gas Distribution System Components
SEMASPEC 91060573B-STD	SEMATECH Test Method for AES Analysis of Surface and Oxide Composition of Electropolished Stainless Steel Tubing for Gas Distribution System Components
SEMASPEC 92071228B-STD	SEMATECH Provisional Test Method for Determining Mass Flow Controller Performance Characteristics from Ambient and Gas Temperature Effects
SEMASPEC 92071229B-STD	SEMATECH Provisional Test Method for Determining Pressure Effects on Indicated and Actual Flow for Mass Flow Controllers
SEMASPEC 92071230B-STD	SEMATECH Provisional Test Method for Determining Steady-State Supply Voltage Effects for Mass Flow Controllers
SEMASPEC 92071231B-STD	SEMATECH Provisional Test Method for Evaluating the Electromagnetic Susceptibility of Thermal Mass Flow Controllers
SEMASPEC 92071232B-STD	SEMATECH Provisional Test Method for Determining Attitude Sensitivity of Mass Flow Controllers (Mounting Position)
SEMASPEC 92071233B-STD	SEMATECH Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers

2.8 SEMI[8]

[8]Semiconductor Equipment and Materials International. 805 East Middlefield Road, Mountain View, CA 94043

This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

3 Terminology

3.1 *Acronyms and Abbreviations*

AES—auger electron spectroscopy

CNC—condensation nucleus counter

CS—conducted susceptibility

EDX—energy-dispersive X ray

EMI—electromagnetic interference

ESCA—electron spectroscopy for chemical analysis

FWHM—full width half maximum

LPC—laser particle counter

MFC—mass flow controller

RS—radiated susceptibility

SEM—scanning electron microscope

STM—scanning tunneling microscope

TOA—take-off angle

3.2 *Descriptions of Terms*

eV—electronvolt

keV—kiloelectronvolt

R_a—roughness average

R_F—fractal-based roughness parameter

4 Significance and Use

The user should consider the result of testing samples when determining the sequence in which tests are performed. For example, if a test of four samples for resistivity results in one sample being unacceptable, then that sample should be rejected. These considerations will help to reduce unnecessary testing. Several samples may be required to run a full set of these tests.

5 Accuracy Test Methods

5.1 *Referenced Documents (Accuracy)*

5.1.0	SEMASPEC #92071221B-STD	SEMATECH Provisional Test Method for Determining Accuracy, Linearity, Repeatability, Short Term Reproducibility, Hysteresis, and Deadband of Thermal Mass Flow Controllers
5.1.1	SEMASPEC #92071222B-STD	SEMATECH Provisional Test Method for Determining Reproducibility and Zero Drift for Thermal Mass Flow Controllers

5.2 *Applicability of Accuracy Test Methods*

5.2.0 SEMASPEC #92071221B-STD, SEMATECH Provisional Test Method for Determining Accuracy, Linearity, Repeatability, Short Term Reproducibility, Hysteresis, and Deadband of Thermal Mass Flow Controllers

This document describes the conditions and procedures for testing the accuracy, repeatability, linearity, hysteresis, and deadband of thermal mass flow controllers (MFCs). Because of the generic nature of the document, all test procedures do not apply to all types of MFCs.

This document also provides a common basis for communication among manufacturers and users.

[**Caution:** The manufacturer's specifications and instructions for installation and operation must be applied during all testing.]

The intent of this document is not to suggest any specific testing program, but only to specify the test methods to be used when testing for parameters that are covered by these methods.

It is not practical to evaluate performance under all possible combinations of operating conditions. The intent of this test procedure, which is run under laboratory conditions, is to collect sufficient data to form a judgment of field performance.

The method of evaluation envisioned is that of checking significant performance characteristics such as accuracy, repeatability, linearity, hysteresis, and deadband under a set of reference operating conditions. Reference operating conditions represent the "best" performance that can be expected.

[**Warning:** When hazardous and toxic gases are used, proper precautions must be taken to ensure safe operation of the system.]

5.2.1 SEMASPEC #92071222B-STD, SEMATECH Provisional Test Method for Determining Reproducibility and Zero Drift for Thermal Mass Flow Controllers

This document describes the conditions and procedures for testing the reproducibility and zero drift of thermal mass flow controllers (MFCs). Because of the generic nature of this document, it does not apply to all types of MFCs.

This document provides a common basis for communication among manufacturers and users.

[**Caution:** It is imperative that the manufacturer's specifications and instructions for installation and operation be applied during all testing.]

The intent of this document is not to suggest any specific testing program, but only to specify the test method to be used when testing for parameters covered by this method.

It is not practical to evaluate performance under all possible combinations of operating conditions. The intent of this test procedure, which is run under laboratory conditions, is to collect sufficient data to form a judgment of field performance.

The method of evaluation envisioned is that of checking reproducibility under a set of reference operating conditions. Reference operating conditions represent the "best" performance that can be expected.

[**Warning:** When hazardous and toxic gases are used, proper precautions must be taken to ensure safe operation of the system.]

6 Performance Test Methods

6.1 *Referenced Documents (Performance)*

6.1.0	SEMASPEC #90120391B–STD	SEMATECH Provisional Test Method for the Determination of the Helium Leak Rate for Gas Distribution System Components
6.1.1	SEMI E17-91	Guideline for Mass Flow Controller Transient Characteristics Tests
6.1.2	SEMASPEC #92071223B-STD	SEMATECH Provisional Test Method for Determining Warmup Time of Mass Flow Controllers
6.1.3	SEMASPEC #92071224B-STD	SEMATECH Provisional Test Method for Determining Reliability of a Mass Flow Controller
6.1.4	SEMASPEC #92071225B-STD	SEMATECH Provisional Test Method for Determining Calibration Accuracy and Calculation of Conversion Factors for a Mass Flow Controller Using Surrogate Gases

6.2 *Applicability of Performance Test Methods*

6.2.0 SEMASPEC #90120391B-STD, SEMATECH Test Method for the Determination of the Helium Leak Rate for Gas Distribution System Components

[Note: Some MFCs are designed to leak across the seat, and an across-the-seat leak test may not be appropriate.]

The purpose of this document is to define a method for testing gas distribution system components being considered for installation into a high-purity gas distribution system. The procedure describes a qualification test method for leak testing using a helium mass spectrometer for ultra–high-purity components of gas distribution systems in semiconductor manufacturing facilities. The method is to be used for gas distribution system components like valves, filters, regulators, and other piping specialties.

This method does not differentiate, however, between mechanical and permeation leak rates. For MFCs, sufficient time must be allowed for permeation effects to be included. A 30 minute minimum exposure to the He leak is recommended. The test must continue until a steady state leak rate is achieved. If steady state is not achieved after three hours, the test is to be terminated, noting that no steady state value was achieved. Steady state is achieved if the baseline leak rate (ignoring spurious peaks) does not vary more than 5% from the existing reading within a 15- minute period. Devices using all metal seals will stabilize quickly while components using elastomeric seals may take over 30 minutes to stabilize.

6.2.1 SEMI E17–91, Guideline for Mass Flow Controller Transient Characteristics Tests

Use this guideline as a test method for characterizing MFC transient and step response with the following modifications:

Replace Section 2.9 (Specified Band) with:

Specified band—the region between ±X% of the final steady state value or ±0.5% of full scale, whichever is greater. The X% is defined during the test procedure as being either 36.8%, 10%, 5%, 2%, 1.0%, 0.5%, and the band specified by the manufacturer's literature for MFC response time.

In Section 3.7 and 3.9, replace 1/5 with 1/4.

In Section 4.4, replace figures 3 and 4 with figures 1 and 2.

In Section 5, replace (4) with a transient plot of indicated flow minus actual flow shall be made. This plot indicates error in the MFC measuring section.

Modify Table 1 as follows:

Initial Setpoint	Off (1)	Off (1)	25	75	Other (3)
Final Setpoint % of Full Scale)	100	25	75	25	OTHER (3)
Dead time (seconds)					
Step Response Time (2%) (Seconds) FS					
Settling time (36.8%) of step					
Settling time (10%) of step					
Settling time (5%) of step					
Settling time (2%) of step					
Settling time (1%) of step					
Settling time (0.5%) of step					
Settling time (MFC Spec)					
Transient Overshoot (Percent of setpoint step change)					
Transient Undershoot (Percent of setpoint step change)					

Transient Response Comments: (Step Response)

Rationale:

Identify MFC time constant (i.e., 63.2% Response Time). The tightened band for steady state response will not give an indication of the quickness of the MFC because of secondary thermal responses.

Various bands can be specified for achieving steady state flow with the MFC. Theoretically specifying a tight band will not change the relative response times between MFCs (assuming V = V’e - t/Tc); however, overtightening the band will make instrumentation difficult and measured response time data scattered. Secondary effects exist which, when superimposed on the primary effect, may be larger than the specified band. In these cases, a quick MFC may achieve 96% response in 0.2 seconds with no overshoot, but it may then take 10 seconds to achieve the last 4%. A slow MFC may achieve only 50% in 1 second and still achieve the final value in 10 seconds. Both MFCs have the same settling time (reference to the tight band) but have markedly different response characteristics. Because of this, we suggest performing the test with the following series of bands:

Response to:	63.2% of step change	(37.8% ±Band)
	90.0%	(10.0%)
	95.0%	(5.0%)
	98.0%	(2.0%)
	99.0%	(1.0%)
	99.5%	(0.5%)
	MFC Stated Spec	Mfr's Specified Band

6.2.2 SEMASPEC #92071223B-STD, SEMATECH Provisional Test Method for Determining Warmup Time of Mass Flow Controllers

This document provides a standardized method for quantifying the warmup time for an MFC.

6.2.3 SEMASPEC #92071224B-STD, SEMATECH Provisional Test Method for Determining Reliability of a Mass Flow Controller

This document describes a method to help determine the ability of an MFC to meet the manufacturer's published specifications over its lifetime. The results of the test will also be useful in the comparison of MFCs.

6.2.4 SEMASPEC #92071225B-STD, SEMATECH Provisional Test Method for Verification of Calibration Accuracy and Calculation of Conversion Factors for a Mass Flow Controller Using Surrogate Gases

This document quantifies the accuracy and linearity of an MFC when mapping an MFC's calibration from one specific gas to another. It also quantifies the flow dependence of an MFC's conversion factor.

7 Contamination Test Methods

7.1 *Referenced Documents (Contamination)*

7.1.0	SEMASPEC #92071226B-STD	SEMATECH Provisional Test Method for Determining Particle Contribution by Mass Flow Controllers
7.1.1	SEMASPEC #92071227B-STD	SEMATECH Provisional Test Method for Determining Moisture, Oxygen, and Total Hydrocarbon Contribution/Retention by Mass Flow Controllers
7.1.2	SEMASPEC #90120400B-STD	SEMATECH Test Method for the Determination of Surface Roughness by Contact Profilometry for Gas Distribution System Components
7.1.3	SEMASPEC #90120401B-STD	SEMATECH Test Method for SEM Analysis of Metallic Surface Condition for Gas Distribution System Components
7.1.4	SEMASPEC #90120402B-STD	SEMATECH Test Method for EDX Analysis of Metallic Surface Condition for Gas Distribution System Components
7.1.5	SEMASPEC #90120403B-STD	SEMATECH Test Method for ESCA Analysis of Surface Composition and Chemistry of Electropolished Stainless Steel Tubing for Gas Distribution System Components
7.1.6	SEMASPEC #90120404B-STD	SEMATECH Test Method for Determination of Surface Roughness by Scanning Tunneling Microscopy for Gas Distribution System Components
7.1.7	SEMASPEC #91060573B–STD	SEMATECH Test Method for AES Analysis of Surface and Oxide Composition of Electropolished Stainless Steel Tubing for Gas Distribution System Components

7.2 *Applicability of Contamination Test Methods*

7.2.0 SEMASPEC #92071226B-STD, SEMATECH Provisional Test Method for Determining Particle Contribution by Mass Flow Controllers

This test method covers MFCs intended for installation into high-purity gas distribution systems. It describes a test method that yields statistically significant comparisons of particulate generation performance among similar MFCs when performed on a specific test apparatus.

A condensation nucleus counter (CNC) or laser particle counter (LPC) is used to measure particle levels.

This document is not intended as a methodology for monitoring in situ particulate performance once a particular MFC has been tested.

The test medium is limited to nitrogen. Actual performance under normal operating conditions may vary.

The accuracy of the data generated by this method is limited to the accuracy of the particle measuring instruments used. The test may not yield comparable results from one test facility to another. It is intended for use by operators who understand the use of the apparatus at a level equivalent to one year of experience.

7.2.1 SEMASPEC #92071227B-STD, SEMATECH Provisional Test Method for Determining Moisture, Oxygen, and Total Hydrocarbon Contribution/Retention by Mass Flow Controllers

The purpose of this document is to define a method for testing MFCs being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among MFCs tested for the purposes of their qualification for installation.

This method establishes a way to test mass flow controllers for moisture, oxygen, and total hydrocarbon contribution/retention within semiconductor gas distribution systems at ambient temperature. In addition, the method allows testing at elevated component temperatures.

This procedure is limited by the sensitivity of instrumentation used, as well as by the response time of that instrumentation.

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

7.2.2 SEMASPEC #90120400B-STD, SEMATECH Test Method for the Determination of Surface Roughness by Contact Profilometry for Gas Distribution System Components

The purpose of this document is to define a method for testing gas distribution system components being considered for installation into a high-purity gas distribution system. Application of this method is expected to yield comparable data among components tested for purposes of their qualification for installation.

The method describes the determination of numerical values for surface roughness. Surface feature sizes from 0.25 μm (10 μin.) R_a to 3.0 μm (120 μin.) R_a are measured using this method.

A statistical analysis of a profilometer graphic output that describes a fractal dimension measurement is included in the Appendix. The resulting numerical value is referred to as the fractal-based roughness, R_F.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Established measurement limits should only be limited by the current technology. For example, surface measurements below 10 R_a μin. may be possible as more advanced styluses become available.

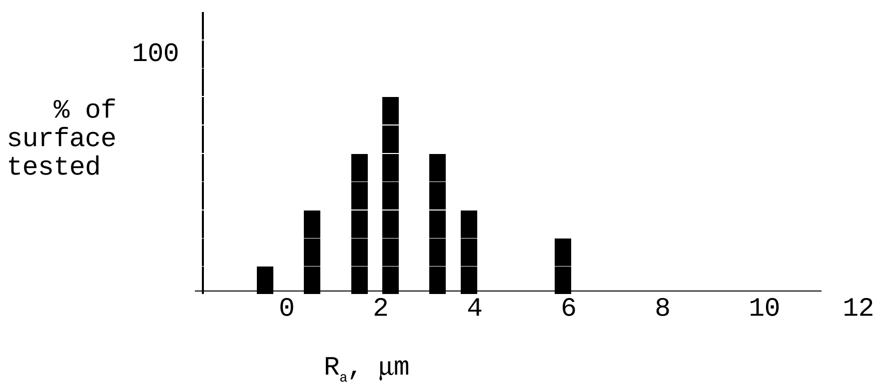
In Section 4.1.6 of the document, contact profilometry measurements should be performed in a Class 100 environment (per FED-STD-209).

This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

In Section 4.4.7, data should be presented as follows:

MFC Surface Roughness

Component Tested	**R_a (µin.)**	**% Surface Area of MFC (tested within the component)**
Body	____________________	__________________%
Sensor Tube	____________________	__________________%
Diaphragm	____________________	__________________%
End Connector	____________________	__________________%
.		
.		
.		
etc.		
Total Surface Area Tested:		______________% (note 1)
Total Surface Area:		______________cm^2



[Note: The total surface area tested will NOT, in most cases, equal 100% of the MFC gas-wetted surfaces.]

7.2.3 SEMASPEC #90120401B–STD, SEMATECH Provisional Test Method for SEM Analysis of Metallic Surface Condition for Gas Distribution System Components

The purpose of this document is to define a method for testing components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for purposes of their qualification for installation.

This document establishes a method of testing interior surfaces of components such as tubing, fittings, and valves for surface morphology.

The technique shall be limited to the assessment of pits, tears, grooves, inclusions, grain boundaries, and other surface anomalies. However, stains and particles that may be produced during specimen preparation should be excluded in the assessment of anomalies.

This procedure applies to all surfaces of tubing, connectors, regulators, valves, and any metal component, regardless of size, and it assumes a scanning electron microscope (SEM) operator skill level typically achieved over a twelve month period.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Regarding Section 4.3.1, since the block of an MFC body is so large, a practical, cost-effective method for sample preparation that will not introduce hydrocarbon contamination does not exist. Therefore, normal metallurgical sample preparation techniques (i.e., use of a cooling lubricant) may be used to prepare the sample. This precludes the use of this test for hydrocarbon contamination analysis.

In Section 4.4.1.1, ASTM Standard E45 should be used for the determination of inclusions.

The comment "[Note: Decreasing the current and re-focusing will increase the quality of the image.]" should be added immediately after Section 4.3.6.

7.2.4 SEMASPEC #90120402B-STD, SEMATECH Test Method for EDX Analysis of Metallic Surface Condition for Gas Distribution System Components

The purpose of this document is to define a method for testing components that are being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for purposes of qualification for this installation.

This document establishes a method for comparing the elemental composition of normal surfaces with any defects found on the surfaces of metal tubing, fittings, valves, or any metal component.

This procedure applies to all metal surfaces of components such as tubing, connectors, regulators, and valves, regardless of size, style, or type, and it assumes an SEM/EDX operator skill level typically achieved over a twelve month period.

The SEM used for this study should conform to those limitations outlined in Provisional SEMASPEC #90120401B-STD and should have a minimum point-to-point resolution of 30 nm.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Regarding Section 4.3.1, since the block of an MFC body is so large, a practical, cost effective method for sample preparation that will not introduce hydrocarbon contamination does not exist. Therefore, normal metallurgical sample preparation techniques (i.e., use of a cooling lubricant) may be used to prepare the sample. This precludes the use of this test for hydrocarbon contamination analysis.

In Section 4.2.2.2, accelerating voltage should be 20 KeV, or as appropriate for elements of interest.

In Section 4.2.2.3, FWHM resolution should be 150 eV or less, rather than 170 eV.

7.2.5 SEMASPEC #90120403B-STD, SEMATECH Test Method for ESCA Analysis of Surface Composition and Chemistry of Electropolished Stainless Steel Tubing for Gas Distribution System Components

The purpose of this document is to define a method for testing components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for the purposes of their qualification for installation.

This document defines a method of testing the interior surface of chromium enhanced stainless steel tubing, fittings and valves, to determine the surface composition and chemistry, as a measure of the effectiveness of electropolishing.

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.

This document describes a test method to characterize "as received" surface composition and chemistry encompassing all chromium enhanced stainless steel surfaces in tubing, connectors, regulators, and valves of all sizes. It describes measurement of Cr/Fe elemental ratios, chemical species ratios for Cr and Fe, and independent estimates of Cr, Fe, and Ni oxide thickness from the electron spectroscopy for chemical analysis (ESCA) methods. Measurement of elemental surface composition and chemistry of phosphorus, sulfur, and residual organic material is also described.

The methodology described assumes the ESCA analyst has a skill level typically achieved with twelve months of experience and a familiarity with the ESCA technique and instrumentation. It is not intended to preclude the use of any particular brand or model of surface analysis equipment. While much of the test methodology has been developed using specific instrumentation, the method can be adapted to most state-of-the-art surface analytical instrumentation. When using this method, it is essential to document the key instrument parameters that define the sampling volume and sensitivity of the measurements.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas-wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas-wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Regarding Section 4.1.2, since the block of an MFC body is so large, a practical, cost-effective method for sample preparation that will not introduce hydrocarbon contamination does not exist. Therefore, normal metallurgical sample preparation techniques (i.e., use of a cooling lubricant) may be used to prepare the sample. This precludes the use of this test for hydrocarbon contamination analysis.

In Section 3.4, the take-off angle (TOA) should be 15° for depths of approximately 20 Å.

7.2.6 SEMASPEC #90120404B-STD, SEMATECH Test Method for Determination of Surface Roughness by Scanning Tunneling Microscopy for Gas Distribution System Components

The purpose of this document is to define a method for testing components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for purposes of their qualification for installation.

Scanning tunneling microscopy (STM) is a noncontact method of profilometry used to measure surface features in the nanometer size range. It can obtain three-dimensional data from a surface, which can then be used to produce a model of the surface texture or to measure surface morphology. The subsequent numerical analysis is to be performed per other standards, such as ANSI B.46.

This method is limited to characterization of stainless steel surfaces that are smoother than $R_a = 0.25$ µm, as determined by a contact-stylus profilometer.

Cutting necessary to obtain access to the areas to be examined must not modify those areas from their condition as found in the component. Etching or conductive coatings of the surface are considered modifications of the gas-wetted surface and are not covered by this test method.

This test method does not cover steels that have an oxide layer too thick to permit tunneling under the test conditions outlined in Section 4.1.

This technique is written with the assumption that the STM operator understands the use of the STM at a level equivalent to one year of experience.

This method assumes that the images obtained are imperturbed by very thin, nonsolid oxide layers (e.g., hydrocarbons, water) on the surface.

The statistical analysis only governs the determination of the R_F value from one-dimensional surface profiles. (See Appendix.) It does not cover those obtained from areas of a surface.

Comparisons between two R_F values are only valid if both values are obtained using the same profiling methods (that is, identical trace lengths, identical scaling of feature heights, and identical means of profile acquisition).

Discussion of tip preparation techniques is outside the scope of this method. Tips can be prepared as described in the literature or as commercially available.

Discussion of artifacts due to tip irregularities, their characterization, and deconvolution from the true surface image is also found in the literature and is outside the scope of this method.

This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas-wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas-wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Other types of non-contact profilometers may be used in lieu of STM. When using an alternative type of non-contact profilometry, the instrument manufacturer's recommended operating procedures should be followed.

7.2.7 SEMASPEC #91060573B-STD, SEMATECH Test Method for AES Analysis of Surface and Oxide Composition of Electropolished Stainless Steel Tubing for Gas Distribution System Components

The purpose of this document is to define a method for testing components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for the purposes of their qualification for installation.

This document defines a method of testing the interior surface of chromium enhanced stainless steel tubing, fittings, and valves to determine the surface composition and chemistry and thereby measure the effectiveness of electropolishing.

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.

This document describes a test method to characterize "as-received" surface composition, oxide composition, and thickness encompassing all interior chromium-enhanced stainless steel surfaces on tubing, connectors, regulators, and valves of all sizes.

This procedure describes the measurement of surface composition of the outer "as-received" surface and a depth composition profile for C, O, Cr, Fe, and Ni in the outer 150 Å of the near-surface oxide film. These measurements quantify the maximum Cr/Fe elemental ratio in the electropolished oxide film and establish a figure-of-merit oxide film thickness in the electropolished surface. This method also describes the measurement of elemental surface composition including P, S, Si, and surface-adsorbed carbon.

This method includes:

1. An initial survey scan at the "as-received" surface to determine composition
2. A depth composition profile to determine elemental distribution and thickness of the oxide layer
3. A survey scan at the end of the profile to determine composition of the bulk material. (This scan is to be done at a depth $\geq$ 1000 Å because the effects of surface modification such as electroplating or other passivation techniques can reach 150 Å, and this scan must reach the bulk material.)

This methodology assumes the AES analyst has a skill level typically achieved with twelve months experience and a familiarity with the AES technique and instrumentation.

This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

The accuracy and precision of all measurements described in this method require that the instrument be calibrated and maintained to all manufacturer's specifications. In addition, sensitivity factors for all pertinent elements must be determined from appropriate reference standards to ensure accuracy and reproducibility of composition data. Calibration procedures are described in detail in Appendix A.1 of the document.

The methodology and instrumentation described in this procedure 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, the method can be adapted to most state-of-the-art surface analytical instrumentation.

Use this method for mass flow controllers with the following modifications:

Specimens that represent the gas-wetted surfaces should be selected. This should include specimens from the body, sensor tube, bypass, end connections, and valve components. A minimum of one test specimen should be examined from each gas-wetted component. The test specimen location should be randomly selected from the surface plane of the component which represents the largest exposed surface area of that component.

Regarding Section 4.1.2, since the block of an MFC body is so large, a practical, cost effective method for sample preparation that will not introduce hydrocarbon contamination does not exist. Therefore, normal metallurgical sample preparation techniques (i.e., use of a cooling lubricant) may be used to prepare the sample. This precludes the use of this test for hydrocarbon contamination analysis.

8 Environmental Test Methods

8.1 *Referenced Documents (Environmental)*

8.1.0	SEMASPEC #92071228B-STD	SEMATECH Provisional Test Method for Determining Mass Flow Controller Performance Characteristics from Ambient and Gas Temperature Effects
8.1.1	EIA RS-414-A	Simulated Shipping Tests for Consumer Electronic Products and Electronic Components
8.1.2	SEMASPEC #92071229B-STD	SEMATECH Provisional Test Method for Determining Pressure Effects On Indicated and Actual Flow for Mass Flow Controllers
8.1.3	SEMASPEC #92071230B-STD	SEMATECH Provisional Test Method for Determining Steady-State Supply Voltage Effects for Mass Flow Controllers
8.1.4	SEMASPEC #92071231B-STD	SEMATECH Provisional Test Method for Evaluating the Electromagnetic Susceptibility of Thermal Mass Flow Controllers
8.1.5	SEMASPEC #92071232B-STD	SEMATECH Provisional Test Method for Determining Attitude Sensitivity of Mass Flow Controllers (Mounting Position)
8.1.6	SEMASPEC #92071233B-STD	SEMATECH Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers

8.2 *Applicability of Environmental Test Methods*

8.2.0 SEMASPEC #92071228B-STD, SEMATECH Provisional Test Method for Determining Mass Flow Controller Performance Characteristics from Ambient and Gas Temperature Effects

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

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 intended to be performed in the following order: Method A—Ambient Temperature Effects (Steady State), Method B—Ambient Temperature Effects (Transient), Method C—Gas Temperature effects (Steady State), Method D—Gas Temperature Effects (Transient). This test method yields the result of setpoint versus actual flow versus indicated flow as influenced by those temperature changes.

This method evaluates MFCs in typically encountered, realistic operating conditions. This test method does not address operational influences outside of the manufacturer's published limitations. This test method is intended for use by operators who understand the use of the apparatus at a level equivalent to one year experience.

8.2.1 EIA RS-414-A, Simulated Shipping Tests for Consumer Electronic Products and Electronic Components

This standard covers simulated shipping tests for consumer electronic products in Section A and simulated shipping tests for electronic components (excluding electronic tubes) in Section B. As simulated shipping tests are developed for other electronic products, they will be added as sections of this standard and will be changed as these revisions occur to reflect the generic content of the standard.

Use this method without qualifications for vibration shock testing of mass flow controllers.

8.2.2 SEMASPEC #92071229B-STD, SEMATECH Provisional Test Method for Determining Pressure Effects on Indicated and Actual Flow for Mass Flow Controllers

The purpose of this document is to define a method for testing MFCs being considered for installation into a high-purity gas distribution system.

This test method measures the upstream (inlet) and downstream (outlet) transient pressure influences on indicated and actual flow. This test method yields the results of actual output flow versus mass flow controller (MFC) set-point and indicated flow as influenced by pressure changes. This test method applies to mass flow controllers with a maximum flow range of 30 slpm. The test methods included herein are: Method A-1—Inlet pressure, step change; Method A-2—Inlet pressure, ramp change; Method B—Outlet pressure, step change.

This test method is limited to analyzing the delta performance characteristics of MFCs and is not a verification of the state of calibration, linearity, or accuracy. This test method does not address pressures in excess of the component's maximum working pressure. All studies will be limited to the specified maximum working pressure of the component. This test method is limited to reasonable pressure transients; i.e., fluctuations otherwise tolerated by common semiconductor process equipment. This test method does not address operational influences outside of the manufacturer's published limitations. This test method does not address interruptions of source gas.

8.2.3 SEMASPEC #92071230B-STD, SEMATECH Provisional Test Method for Determining Steady-State Supply Voltage Effects for Mass Flow Controllers

The purpose of this document is to define a method of testing MFCs for steady-state supply voltage effects on the MFC's ability to accurately deliver setpoint flow values.

This procedure applies to thermal MFCs. It is intended to measure the delivered mass flow rate variation as a function of deviation from the reference steady-state supply voltage. The test method is designed for DC-powered MFCs. The supply voltage effects include voltage depression and over-voltage variations in the DC supply.

This test method is not designed for AC-powered MFCs. This test method addresses steady-state effects, not effects caused by transient power supply behavior. This method is written with the assumption that the operator understands the use of the apparatus at a level equivalent to one year of experience.

8.2.4 SEMASPEC #92071231B-STD, SEMATECH Provisional Test Method for Evaluating the Electromagnetic Susceptibility of Thermal Mass Flow Controllers

A thermal MFC should be designed and assembled so that it is compatible with the electromagnetic environment in which it is to be used. This document presents a test method that may be applied to evaluate the susceptibility of the controller electronics to electromagnetic interference (EMI).

The purpose of this document is to define a structured method for testing and evaluating the electromagnetic susceptibility of thermal mass flow controllers.

This document contains the requirements and test method that can be used to ensure that a thermal MFC will maintain its functional characteristics when subjected to EMI levels typical of the industry. The test method covers both the radiated susceptibility (RS) and conducted susceptibility (CS) of the controller when exposed to EMI. The electromagnetic susceptibility requirements are extracted from MIL-STD-461C and SAMA PMC-33.1, and the test method is a culmination of the RS03, CS01, CS02, and CS06 test methods defined in MIL-STD 462.

8.2.5 SEMASPEC #92071232B-STD, SEMATECH Provisional Test Method for Determining Attitude Sensitivity of Mass Flow Controllers (Mounting Position)

The purpose of this test is to determine the attitude sensitivity of various MFCs. Attitude refers to the physical orientation of the MFC. There are two orientations of interest, one with the direction of flow horizontal to the surface of the earth and the other with the direction of flow perpendicular to the surface of the earth.

The MFC is operated during this test and changes in calibration are monitored.

This test is limited to measuring the change in performance of the MFC and is not intended to verify the accuracy of the original calibration of the device.

8.2.6 SEMASPEC #92071233B-STD, SEMATECH Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers

MFCs are often used to control corrosive gases under unfavorable conditions. This test method is intended to help differentiate between MFC designs on the basis of relative resistance to corrosion-induced failure.

This test method describes a corrosive gas exposure test for MFCs. It is intentionally aggravated to accelerate the test while simulating conditions that may be found within process equipment gas systems in the semiconductor industry. The MFC is operated during the test, and corrosion is indirectly detected by observing changes in MFC calibration and other operating parameters.

This test is intended for MFCs manufactured for use in hydrogen chloride or in a similar, highly corrosive gaseous environment.

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