

SEMI F104-0520

TEST METHOD FOR EVALUATION OF PARTICLE CONTRIBUTION OF COMPONENTS USED IN ULTRAPURE WATER AND LIQUID CHEMICAL DISTRIBUTION SYSTEMS

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NOTICE: This Document was completely rewritten in 2019.

1 Purpose

1.1 This Document provides specifications and test methods to evaluate and compare components for particle contribution into an ultrapure water (UPW) fluid stream. Two methods for evaluating particle contribution are provided.

1.1.1 The rinse test method evaluates performance in a flushing mode while the component is in a static state (no actively moving components within the component under test). It provides an indication of the flush volume required to bring particle levels to specification.

1.1.2 The dynamic test method evaluates performance during component operation and provides an indication of the steady state particle contribution due to dynamic operating conditions.

2 Scope

2.1 These test methods apply to liquid chemical and UPW system components intended for use in semiconductor manufacturing tools and ancillary equipment.

2.2 This Document describes methods for measuring particle contribution from components while using UPW as the test media. UPW, for the intents and purposes of this Document, is defined as having the minimum requirements as outlined in ¶ 7.4 of this Document.

2.3 These methods use an in situ liquid optical particle measurement (OPM) instrument to quantify performance.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 These methods specify flow rates to enhance the particle measurement sensitivity of the test and to standardize testing conditions. Actual performance under normal operating conditions may vary.

3.2 These methods are limited to testing components with an orifice size from 3.175 mm (1/8 inch) minimum to approximately 63 mm (2 inch) maximum.

3.3 The accuracy of the data generated by these methods is limited to the accuracy of the OPM utilized.

3.4 Results of these methods are a relative measurement. Results can differ between OPMs due to differences in manufacturers' calibration procedures, differences in relative response to naturally occurring particle types, and differences in OPM backgrounds. The user should be aware that results from different OPMs may be difficult to compare without considerable effort to correlate results. The user should also be aware that results from different systems, although using the same OPM, may be difficult to correlate.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI E49 — Guide for High Purity and Ultrahigh Purity Piping Performance, Subassemblies, and Final Assemblies

SEMI F57 — Specification for Polymer Components Used in Ultrapure Water and Liquid Chemical Distribution Systems

4.2 Federal Standards¹

FED STD 209E — Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones

4.3 ISO Standards²

ISO 14644-1:1999 — Cleanrooms and Associated Controlled Environments – Part 1: Classification of Air Cleanliness

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *FM* — flow meter

5.1.2 *OPM* — optical particle measurement instrument (This includes optical particle counters, optical particle monitors, and optical particle spectrometers [see Figure 1].)

5.1.3 *P* — pressure transducer or pressure gauge (See P1 and P2 in Figure 1. The pressure transducers or gauges shall have an error of $\leq 2.5\%$ of reading.)

5.1.4 *UPW* — ultrapure water

5.1.5 *V* — manual or pneumatic valve (See Figure 1, which uses manual valves for V1 through V4.)

5.1.6 *VR* — variable restrictor (See Figure 1.)

5.2 Definitions

5.2.1 *arithmetic average* — method of averaging defined as:

$$AA_y = (y_1 + y_2 + \dots + y_n) / n \quad (1)$$

where:

y_i = particular value in the series, and

n = number of values in the series.

5.2.2 *bypass FM* — Flow meter (FM) for measuring the bypass flow rate. This device may have the capability of controlling flow as well. This FM should have accuracy within $\pm 2.5\%$ over the flow rate range of interest (see Figure 1).

5.2.3 *bypass flow rate* — that portion of the total flow rate that bypasses the OPM (see Bypass FM in Figure 1).

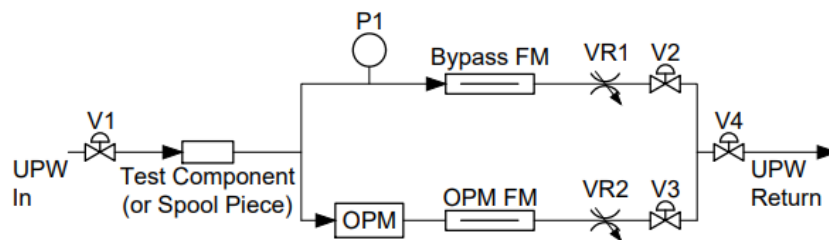


Figure 1
Generic Test Schematic (For Terminology Only)

¹ Federal Communications Commission, 445 12th St. S.W., Washington DC 20554; Telephone: +1.888.225.5322, <http://www.fcc.gov>

² International Organization for Standardization, ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; Telephone: +41.22.749.01.11, <http://www.iso.org>

5.2.4 *counting interval* — the time increment over which the number of particles is recorded.

5.2.5 *dynamic flush* — a condition where the moving parts within the component's wetted flow path are intentionally cycled or are known to move due to changing operating conditions.

5.2.5.1 Examples of dynamic flushing include:

- Fully opening and fully closing a pneumatic, manual, or electronic valve. The valve has to open and close one time for a cycle.
- Varying the fluid inlet pressure to a regulator while maintaining a constant downstream pressure.

5.2.6 *geometric average* — method of averaging defined as:

$$GAy = (y1 \times y2 \times \dots \times yn)^{(1/n)} \quad (2)$$

where:

y_i = particular value in the series, and

n = number of values in the series.

5.2.7 *OPM FM* — FM for measuring the OPM flow rate. This may be built within the OPM and may have the capability of controlling flow as well. It should have a flow rate range and accuracy corresponding to the OPM manufacturer's recommendations. In addition, this device must be downstream of the OPM sensor to avoid contributing particles to the UPW being measured (see Figure 1).

5.2.8 *OPM flow rate* — that portion of the total flow rate that flows through the OPM and is measured by the OPM FM (see OPM FM in Figure 1).

5.2.9 *OPM particle size channel* — an instrument defined bin used to accumulate the counts of particles. Typically, channels can refer to either cumulative (i.e., particles ≥ 30 nm) or differential particles (i.e., ≥ 30 nm but < 50 nm).

5.2.10 *particle* — for the purposes of this method, an ensemble of molecules large enough to be detected by the analytical method used to detect it. The corresponding size of the particle is defined by the analytical method used. For example, when particles are sized using a laser optical particle counter, the size of the particle is determined by its scattering cross section. An equivalent particle diameter is calculated based on an OPM calibration using Polystyrene Latex particles.

5.2.11 *sample flow rate* — that portion of the OPM flow rate that is examined by the OPM sensor.

5.2.12 *spool piece* — a straight connecting piece with similar end connections and orifice size to the component to be tested. This is used in place of the component to establish the test system background level prior to installing the component under test.

5.2.13 *spool piece installation cleanup curve* — a plot of particle concentration versus volume passed through the spool piece, immediately after removing the spool piece, draining it, and immediately reinstalling it in the system.

5.2.14 *static flush state* — a condition where the moving parts within the component's wetted flow path are not intentionally cycled.

5.2.15 *test pressure* — pressure downstream of the components under test after the split to the OPM (see P1 in Figure 1).

5.2.16 *test system background* — the average particle concentration in the test system reported by the OPM when flowing the UPW through a spool piece, after rinsing to a steady state particle concentration. This includes contributions from the UPW, OPM, and the test system.

5.2.17 *test resistivity* — the resistivity of the UPW as measured on the facilities supply loop. This is monitored to ensure that the UPW is in accordance with ¶ 7.4.

5.2.18 *test temperature* — the temperature of the UPW as measured on the facilities supply loop. This is monitored to ensure that the UPW is in accordance with ¶ 7.4.

5.2.19 *total flow* — the combined flow rate through the bypass FM and OPM FM.

6 Requirements

6.1 Testing Requirements

6.1.1 Components requiring particle testing are listed in Table 1.

Table 1 Testing Requirements

Type	Particles	
	Rinse Test	Cycle/Dynamic Test
Pneumatic Valves	√	√
Electronic Valves	√	√
Manual Valves	√	-
Flow Meters: No moving parts	√	-
Flow Meters: With moving parts	√	√
Flow Controllers	√	√
Pneumatic Pilot Regulators	-	√
Manual Regulators	-	√
Tubing	√	-
Degas Modules/large surface area contactors	TBD	TBD

6.2 Particle Contribution Requirements

6.2.1 *Importance of Test* — Particles released from within polymer components can come into direct contact with microelectronic devices and other critical surfaces causing unwanted surface contamination or disrupting the photolithography process, thereby decreasing yields.

6.2.2 Particle contribution testing uses methods for measuring particle contribution from components while using UPW as the test medium. These methods use an in situ OPM instrument to quantify performance.

6.2.3 Particle contribution testing is restricted to polymer components as shown in Table 1.

6.2.4 Components shall conform to the particle contribution specifications appearing in Tables 2 through 5.

NOTE 1: The performance values in Tables 2 through 5 for the rinse test and cycle test represent geometric and arithmetic averages, respectfully, from a minimum of four representative components after subtracting an initial system particle background level.

Table 2 Valves and Flow Controllers

Static Rinse Specification				Dynamic Rinse Specification	
Orifice Size (mm)	Orifice Size (inch)	Total Flow Rate (mL/min)	Maximum volume to reach ≤ 10 particles added per liter ≥ 50 nm (Liters)	Test Flow Rate (mL/min)	Maximum particles added per actuation ≥ 50 nm after 500 and 10,000 cycles (#)
3–4	$\frac{1}{8}$	300	500	250	10
6–7	$\frac{1}{4}$	600	500	250	10
9–10	$\frac{3}{8}$	900	1000	250	50
12–13	$\frac{1}{2}$	1200	1000	250	100
19–20	$\frac{3}{4}$	1800	2500	500	200
25–26	1	2400	2500	500	200
32	1.25	3000	5000	1000	500
40	1.25				

Static Rinse Specification				Dynamic Rinse Specification	
Orifice Size (mm)	Orifice Size (inch)	Total Flow Rate (mL/min)	Maximum volume to reach ≤ 10 particles added per liter ≥ 50 nm (Liters)	Test Flow Rate (mL/min)	Maximum particles added per actuation ≥ 50 nm after 500 and 10,000 cycles (#)
50	1.5	3500	5000	1000	1000
63	2	5000	10000	1000	1000

Table 3 Tubing

Static Rinse Specification			
Tubing OD (mm)	Tubing OD (inch)	Total Flow Rate (mL/min)	Spec (Liters to reach < 10 particles > 50 nm added per liter per 10 meters)
6	0.25	600	1000
10	0.375	1350	2500
13	0.5	1800	5000
19	0.75	2700	7500
25	1	3600	10000
32	1.25	4500	10000
38	1.5	5400	10000

Table 4 Flow Meters

Static Rinse Specification			
Orifice Size (mm)	Orifice Size (inch)	Total Flow Rate (mL/min)	Maximum volume to reach ≤ 10 particles added per liter ≥ 50 nm (Liters)
3–4	$\frac{1}{8}$	300	500
6–7	$\frac{1}{4}$	600	500
9–10	$\frac{3}{8}$	900	1000
12–13	$\frac{1}{2}$	1200	1000
19–20	$\frac{3}{4}$	1800	2500
25–26	1	2400	2500
32	1.25	3000	5000
40	1.25		
50	1.5	3500	5000
63	2	5000	10000

Table 5 Regulators

<i>Dynamic Rinse Specification</i>			
<i>Orifice Size (mm)</i>	<i>Orifice Size (inch)</i>	<i>Test Flow Rate (mL/min)</i>	<i>Number of particles added per liter ≥ 50 nm after 48 hours of testing</i>
6–7	$\frac{1}{4}$	900	Report
9–10	$\frac{3}{8}$	1350	Report
12–13	$\frac{1}{2}$	1800	Report
19–20	$\frac{3}{4}$	2700	Report
25–26	1	3600	Report
32	1.25	4500	Report
40	1.25		Report
50	1.5	5400	Report
63	2	7200	Report

6.2.5 This Document defines the test method to be used when conducting particle contribution testing.

6.2.6 The static rinse test evaluates particle contribution performance in a flushing mode while the component is in a static state. It provides an indication of the volume of UPW required to bring particle levels to the guidelines defined in Tables 2 through 5.

6.2.7 The dynamic rinse test evaluates particle contribution performance during active operation of the component such as cycling a valve on and off or subjecting a regulator to dynamic inlet pressure conditions.

7 Test Stand Configuration

7.1 The test system must be enclosed in an ISO Class 7 (per current revision of ISO 14644-1:1999, roughly equivalent to FED STD 209E Class 10,000) or better environment. Some OPMs may have more stringent requirements for their environment and those requirements should be met. Procedures necessary to maintain ISO Class 7 or better when handling any part of the test system or during testing must be followed.

7.2 Ultrahigh purity, low particle piping systems must be used in all wetted flow paths from the UPW source to the OPM to ensure that the requirements of ¶ 7.4 are maintained. The recommendations within SEMI E49 should be considered when designing and assembling the system.

7.3 Protect the test system from excessive vibration which could lead to high particle background counts.

7.4 A source of water is required which complies with the following minimum requirements:

7.4.1 Background particle concentration ≤ 0.2 particles/mL (≥ 50 nm) Temperature of $25 \pm 5^\circ\text{C}$ ($77 \pm 9^\circ\text{F}$).

7.4.2 Resistivity $\geq 18 \text{ M}\Omega\cdot\text{cm}$ at 25°C (77°F).

7.5 Components must be prepared and installed in accordance with manufacturers' recommendations prior to the onset of testing.

7.6 Operating pressure into the OPM (see P1 in Figure 1) must be $200 \pm 35 \text{ kPa}$ ($30 \pm 5 \text{ psig}$) during testing.

7.7 Ensure that the OPM, FMs, and pressure gauges or transducers are calibrated in accordance with manufacturers' procedures and frequency of calibration.

7.8 OPMs can give erroneous results if bubbles are present. The components under test, the plumbing between the components under test, and the OPM should be oriented to limit the likelihood of bubble entrapment. The test components should be positioned such that the wetted path is in a vertical orientation.

7.9 A data acquisition system is required for the OPM. The data acquisition system must be able to capture particle data in the particle size channels of principle interest at least once per minute.

7.10 A means of actuating the component under test is necessary when performing the cycle test method.