

SEMI F104-1107

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

This standard was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on September 5, 2007. It was available at www.semi.org in October 2007.

1 Purpose

1.1 This document provides recommended test methods to evaluate and compare components for particle contribution into an Ultrapure Water (UPW) fluid stream. These methods fulfill the particle testing requirements within SEMI F57.

1.2 Two methods for evaluating particle contribution are provided.

1.2.1 The rinse test method evaluates performance in a flushing mode while the component is in a static state. It provides an indication of the volume required to bring particle levels to a user-defined specification.

1.2.2 The cycle test method evaluates performance during component cycling and provides an indication of the steady state particle contribution due to cycling.

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. Components consisting of, but not limited to, those listed in Table 1 can be evaluated with the test methods indicated.

NOTE 1: These methods are not recommended for filters or pumps.

Table 1 Testing Requirements

<i>Component Type</i>	<i>Rinse Test</i>	<i>Cycle Test</i>
Pneumatic Valves	X	X
Electronic Valves	X	X
Manual Valves	X	X
Check Valves	X	X
Flow Meters	TBD	TBD
Flow Controllers	TBD	TBD
Pneumatic Pilot Regulators	X	X
Manual Regulators	X	X
Tubing	X	N/A
Filter Housings	X	N/A
Degas Modules	X	N/A
Transducers	X	N/A
Gauge Guards	X	N/A
Fittings	X	N/A

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 ¶ 6.4 of this document.

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

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

3 Limitations

3.1 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 1/8" (3.175 mm) minimum to 1" (25.4 mm) 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 OPM's 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 OPM's 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

SEMI E49.7 — Purity Guide for the Design and Manufacture of Ultrapure Water and Liquid Chemical Systems in Semiconductor Process Equipment

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

4.2 Federal Standard¹

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

4.3 ISO Standard²

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

5 Terminology

5.1 Abbreviations and Acronyms

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

5.1.2 *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.

5.1.3 *UPW* — Ultrapure Water

5.1.4 *V* — manual or pneumatic valve (see Figure 1, which uses manual valves for V1–V4).

5.1.5 *VR* — variable restrictor (see Figure 1).

5.2 Definitions

5.2.1 *Arithmetic Average* — method of averaging defined as:

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

¹ Available from General Service Administration, Federal Supply Service Bureau, Specification Section, Suite 8167, 470 East L'Enfant Place SW, Washington, D.C. 20407.

² International Organization for Standardization, ISO Central Secretariat, 1 rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland. Telephone: 41.22.749.01.11; Fax: 41.22.733.34.30; <http://www.iso.ch>

where:

y_i = particular value in the series
 n = number of values in the series

5.2.2 *Geometric Average* — method of averaging defined as:

$$GA_y = (y_1 \times y_2 \times \dots \times y_n)^{(1/n)} \quad (2)$$

where:

y_i = particular value in the series
 n = number of values in the series

5.2.3 *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.

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

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

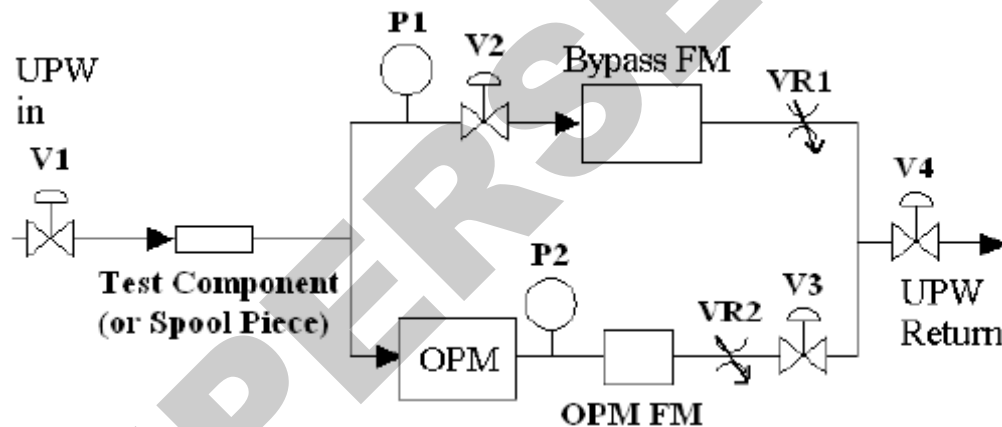


Figure 1
Generic Test Schematic (For Terminology Only)

5.2.6 *counting interval* — the time increment over which the number of particles are recorded.

5.2.7 *cycling* — actuation of a component with moving parts within the wetted flow path. Examples of cycling include:

- Fully opening and fully closing a pneumatic, manual, or electronic valve. The valve has to open and close one time for a cycle.
- Starting and stopping flow through a check valve via a downstream valve. The check valve has to open and close one time for a cycle.

5.2.8 *OPM FM* (see Figure 1) — flow meter 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 should be downstream of the OPM sensor to avoid contributing particles to the UPW being measured.

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 $\geq 0.1 \mu\text{m}$) or differential particles (i.e. $\geq 0.1 \mu\text{m}$ but $\leq 0.15 \mu\text{m}$).

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

5.2.11 *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.

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

5.2.13 *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.

5.2.14 *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.

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

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

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 ¶ 6.4.

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 ¶ 6.4.

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

6 Test Stand Configuration

6.1 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 OPM's 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.

6.2 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 ¶ 6.4 are maintained. The recommendations within SEMI E49.7 should be considered when designing and assembling the system.

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

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

6.4.1 Operating pressure downstream of the OPM (see P2 in Figure 1) must be ≥ 20 psig (138 kPa) during testing.

6.4.2 Particle concentration ≤ 0.05 particles/ml ($\geq 0.1 \mu\text{m}$) OR $\leq 10\%$ of user defined performance specification, whichever is greater.

6.4.3 Temperature of $25 \pm 5^\circ\text{C}$ ($77 \pm 9^\circ\text{F}$).

6.4.4 Resistivity ≥ 18 megaohm-cm @ 25°C (77°F).

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

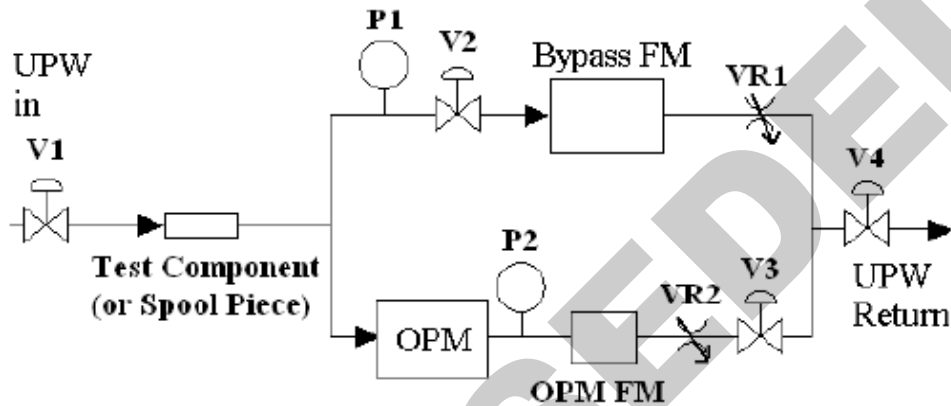
6.6 Ensure that the OPM, flow meters, and pressure gauges or transducers are calibrated in accordance with manufacturers' procedures and frequency of calibration.

6.7 OPM's 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.

6.8 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.

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

6.10 For system background level testing, configure the test stand in accordance with Figure 2 and insert a spool piece. For rinse testing, configure the test stand in accordance with Figure 2 and insert a test component or test assembly.



NOTE: This schematic is also used for cycle testing manual valves and manual regulators.

Figure 2
Background/Rinse Test Schematic

6.11 For cycle testing components such as valves and regulators, configure the test stand in accordance with Figure 3.

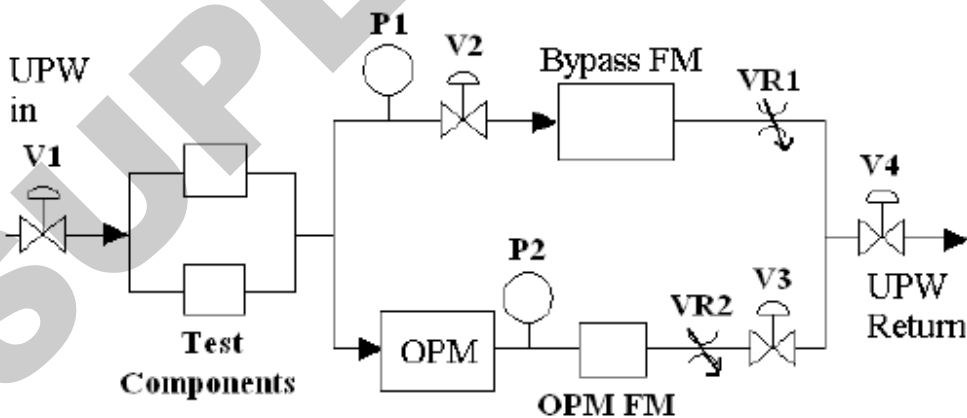


Figure 3
Valve/Regulator Cycle Test Schematic

6.12 For cycle testing check valves only, configure the test stand in accordance with Figure 4.

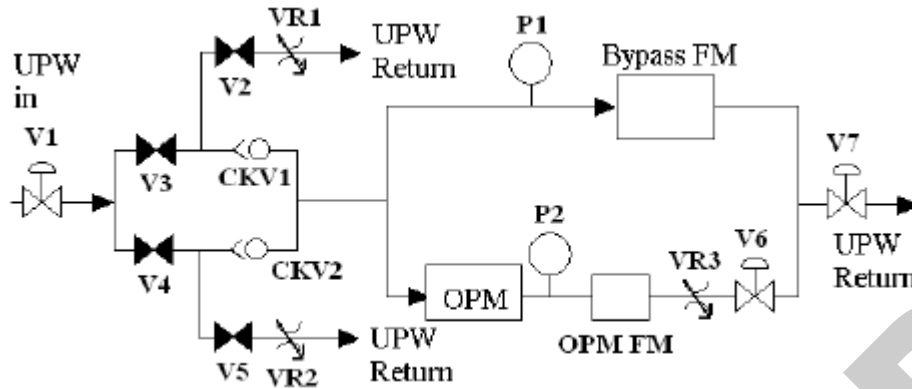


Figure 4
Check Valve Cycle Test Schematic

7 Sample Recommendations

- 7.1 A representative sample of the population of components under interest should be selected for testing.
- 7.2 For components with very low particle levels it may be necessary to place several components in series as an assembly to be able to quantify particle contribution above the background level.
- 7.3 A minimum of 4 components or component assemblies should be tested to assess variability. If more than 4 are to be tested, it is recommended that the sample size be an even number as most of the cycle test procedures are performed with component pairs.
- 7.4 Component handling and packaging may impact particle test results. When testing, component handling and packaging should be in accordance with the typical manufacturing processes.

8 Procedures

8.1 General Procedures

- 8.1.1 Throughout this document, the steps referring to the starting and stopping of UPW flow should be carried out by using valves located at UPW In and UPW Return as shown in Figures 1–4.
- 8.1.2 The steps referring to monitoring and adjusting the flow should be carried out using the OPM flow meter and the bypass flow meter shown in Figures 1–4. The flow through the OPM flow meter should remain constant as specified by the OPM manufacturer.
- 8.1.3 This document uses the following progression for measuring particle contribution into a UPW fluid stream.

¶ 8.2: *System Background Level Test* — establishes background level of the system to discern the particle contribution from the test stand versus the components under test.



¶ 8.3: *Spool Piece Installation Rinse Test* — quantifies the impact of installing a clean spool piece into the test system.



¶¶ 8.4–8.5: *Rinse Test* — measures the particle concentration of the UPW fluid stream after passing through the component under test in a static state.



¶¶ 8.6–8.10: *Cycle Test (if applicable)* — measures the particle concentration of the UPW fluid stream after passing through the component under test while cycling.

