

SEMI C82-0713 (Reapproved 0819) TEST METHOD FOR PARTICLE REMOVAL PERFORMANCE OF LIQUID FILTER RATED 20 TO 50 NM WITH LIQUID-BORNE PARTICLE COUNTER

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1 Purpose

1.1 This Document is to provide a standard of monodispersed gold nanoparticle (GNP) challenge test for liquid filter rated 20 to 50 nm using optical liquid-borne particle counter.

2 Scope

2.1 This Document covers a monodispersed GNP challenge test method for 20 to 50 nm rated liquid filter.

2.2 This Document defines a test condition for monodispersed GNP challenge test.

2.3 The following areas are to be addressed in this Document:

- The test condition such as fluid, flow rate, pressure, GNP concentration, ligand concentration, membrane treatment etc.
- The type of filter evaluated.
- The type of GNP (reference material).
- The method of membrane treatment prior to the test for decreasing the adsorbing effect.
- The description of the test result.

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 The test procedure is destructive; the filter cannot be returned into operation.

3.2 Material purity and potential leaching of dissolved contaminants are not addressed by this Standard. SEMI F57 does not address this issue for final filters therefore the end user may decide to consider such testing when selecting different types of filters.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI F63 — Guide for Ultrapure Water Used in Semiconductor Processing

SEMI F110 — Test Method for Mono-dispersed Polystyrene Latex (PSL) Challenge of Liquid Filters

NOTE 1: The SEMI NA Liquid Chemicals Committee is developing Evaluation of the Efficacy of the Filters of Ultrapure Water Distributions Systems intended to be used in conjunction with this Document.

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 *GNP* — gold nanoparticle

- 5.1.3 *LPM* — liter per minute (L/min)
- 5.1.4 *LRV* — log reduction value
- 5.1.5 *MSA* — Mercaptosuccinic acid
- 5.1.6 *NIST* — National Institute of Standards and Technology
- 5.1.7 *OPC* — optical particle counter
- 5.1.8 *P* — pressure gauge
- 5.1.9 *PSL* — polystyrene latex
- 5.1.10 *T* — thermometer
- 5.1.11 *UPW* — ultrapure water

5.2 Definitions

- 5.2.1 *background* — the number of OPC count when feeding the water without GNP.
- 5.2.2 *challenge* — the feed the water including GNP and ligand to test filter.
- 5.2.3 *efficiency* — particle removal efficiency of filter measured by this Test Method. It is the effectiveness of the filter in removing the particles, and is measured as $(\text{Upstream} - \text{Downstream}) / \text{Upstream} \times 100$.
- 5.2.4 *ligand* — ion or molecule (chemicals) that could bind with the surface of gold nanoparticle.
- 5.2.5 *log reduction value (LRV)* — log reduction value of filter measured by test method. This is measured as logarithmic value of ratio of upstream to downstream particle counts.

6 Summary of Test Method

6.1 This Test Method describes that test equipment and procedures for determining the particle removal efficiency of liquid filter (20 to 50 nm) with the water including ligand and GNP by calculating the difference of the number of challenged particle between upstream and downstream. The number of challenged particle is counted with optical particle counter.

7 Apparatus

7.1 Test Device

- 7.1.1 For this Test Method, use the schematic shown in Figure 1 or Figure 2.
- 7.1.2 The standard test device shall consist of a prefilter, flow meters, a particle injection device, pressure gauges, a test filter, a particle counter, flow control valves, a resistivity sensor, a thermometer, and tubing connecting them.
- 7.1.3 The pore size of a prefilter shall be equal to or smaller than the pore size of a test filter.
- 7.1.4 Use a flow meter with an allowable error to less than 5% full scale and of appropriate range.
- 7.1.5 Use a pressure gauge with an allowable error to less than 1% full scale and of appropriate range.
- 7.1.6 Use a thermometer with an allowable error to less than 1% full scale and of appropriate range.
- 7.1.7 Use a tubing with an inner diameter that does not lead to significant pressure loss for the fluid medium used in the test.

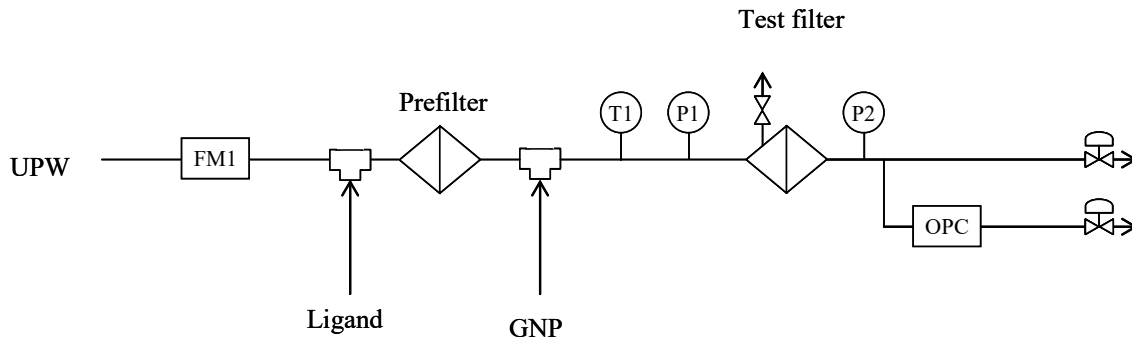


Figure 1
Schematic of Typical Test Setup (Example 1)

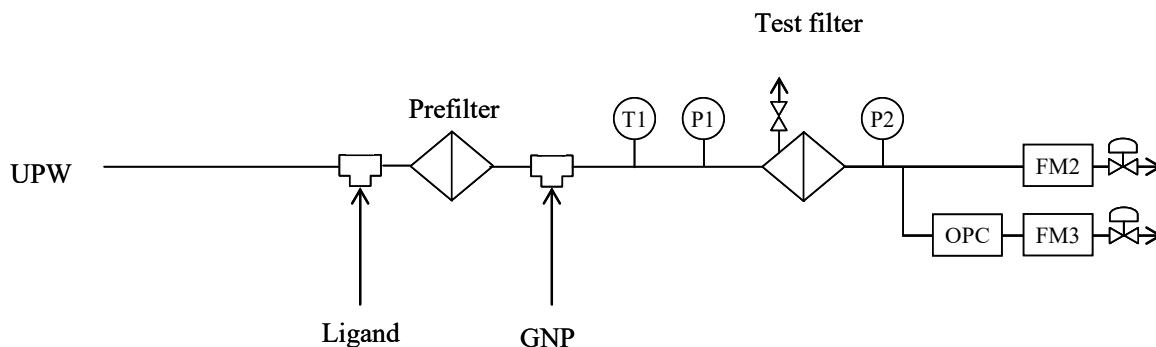


Figure 2
Schematic of Typical Test Setup (Example 2)

7.2 Particle Injection Device

7.2.1 Use a particle injection device that can inject a particle at a constant flow rate without a pulsation.

7.3 Ligand Injection Device

7.3.1 Use a ligand injection device that can inject a ligand at a constant flow rate without a pulsation.

7.4 Particle Counter

7.4.1 Use the light scattering method particle counter.

7.4.2 Use a particle counter capable of measuring particle size to smaller than test GNP size.

7.4.3 Use cumulative counts of above minimum measurable particle size with OPC as count value.

NOTE 2: The intensity of scattered light from GNP is approximately 2.5 times of the one of polystyrene latex particle. Accordingly, OPC could detect smaller GNP than PSL. For example, the OPC capable to detect 50 nm particle (calibrated with 50 nm PSL), could detect 20 nm-sized GNP (see Appendix 2).

8 Reagents and Materials

8.1 Ultrapure Water (UPW)

8.1.1 Use the supply UPW with the resistivity more than 17 MΩ·cm.

8.1.2 Temperature of the UPW shall be adjusted to 25 ±5°C.

NOTE 3: As some ligands or other surfactants are added to actual challenge solution, there is no need to adhere to the resistivity described in ¶ 8.1.1 at challenge test.

8.2 GNP

8.2.1 Use the monodispersed GNP, whose size is as same as the GNP certified by NIST.

8.2.2 The monodispersed GNP with 20 to 50 nm of particle size is used.

8.3 Ligand

8.3.1 Use a ligand to make colloidal GNP stably disperse and decrease the interaction between GNP and filter media (see Appendix 1).

9 Test Specimens

9.1 Use a liquid filter from rating 20 to 50 nm.

9.2 Any size and type of filter, such as disk, capsule or cartridge, can be used in the test.

10 Procedure

10.1 General Procedures

10.1.1 The filter is evaluated by the following procedures:

- Testline background
- Filter pretreatment and filter flushing
- Background
- Challenge

10.2 Testline Background

10.2.1 Background testing is required for every test before installing the test filter into the test line (see Figure 1 or Figure 2).

10.2.2 Set the test condition (flow rate and pressure):

- UPW (Resistivity: $\geq 17 \text{ M}\Omega\cdot\text{cm}$, temperature: $25 \pm 5^\circ\text{C}$)
- FM1 or FM2 + FM3
 - Choose either standard below:
 1. Filter size standard: $n \text{ L/min}$ at $25.4 \times n \text{ mm}$ -sized ($n \text{ inch}$ -sized) filter, ($n = 1, 2, 3, 4, 5 \dots$)
 2. Filter media surface area standard: the flow rate to achieve the flux of 1 mL/min/cm^2 .
- P2: $\geq 100 \text{ kPa}$

NOTE 4: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

10.2.3 Start measurement using the OPC. It is necessary to count and confirm the stable values for more than 30 minutes.

10.2.4 Record particle levels in at least ten minute intervals.

10.2.5 Continue the particle measurement until the OPC counts becomes less than 10 counts/mL ($\geq 50 \text{ nm}$).

10.3 Filter Pretreatment

10.3.1 Prewetting is needed if the test filter is hydrophobic (follow filter manufacturer's instructions).

10.3.2 Pretreatment fluid is aqueous solution of a ligand used at the challenge test. The concentration shall be same with the concentration at the challenge test.

NOTE 5: The condition of ligand concentration to achieve the lowest adsorbing effect depends on membrane type. Accordingly, it is necessary to optimize it in advance. Please see Appendix 1 in terms of how to set the ligand condition.

10.3.3 Fill the membrane pore with the pretreatment fluid by flowing after installing into the test line.

10.3.3.1 After ¶ 10.3.3, soak filters in the pretreatment fluid for a minimum of 30 minutes.

10.4 Background

10.4.1 Start UPW flow and ligand injection.

10.4.2 Count the particles at downstream of the filter with the OPC before the challenge testing.

10.4.3 Close the air-vent valve after venting air from the filter upstream.

10.4.4 Set the test condition (flow rate and pressure).

- UPW (Resistivity: $\geq 17 \text{ M}\Omega\cdot\text{cm}$, temperature: $25 \pm 5^\circ\text{C}$)
- FM1 or FM2 + FM3

Choose either standard below:

- (1) Filter size standard: $n \text{ L/min}$ at $25.4 \times n \text{ mm}$ -sized ($n \text{ inch}$ -sized) filter, ($n = 1, 2, 3, 4, 5 \dots$)
- (2) Filter media surface area standard: the flow rate to achieve the flux of 1 mL/min/cm^2 .

- P2: $\geq 100 \text{ kPa}$

NOTE 6: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

10.4.5 Start measurement of OPC.

10.4.5.1 Record particle levels in at least ten minute intervals.

10.4.5.2 Continue the particle measurement until the OPC counts become less than 10 counts/mL ($\geq 50 \text{ nm}$) for more than 30 minutes.

NOTE 7: If the OPC counts do not become less than 10 counts/mL, terminate the challenge test and re-check test line and other apparatuses. Then, restart the challenge test from § 10.4.

10.5 Challenge

10.5.1 Prepare the challenge monodispersed GNP When the challenge solution is diluted, it is necessary to use UPW characterized at ¶ 8.1 and agitate sufficiently. The prepared challenge solution shall be used in the test within 24 hours.

10.5.2 Install the particle counter directly downstream of the test filter (see Figure 1 or Figure 2).

10.5.3 Set the test condition (flow rate and pressure).

- FM1 or FM2 + FM3

Choose either standard below:

- (1) Filter size standard: $n \text{ L/min}$ at $25.4 \times n \text{ mm}$ -sized ($n \text{ inch}$ -sized) filter, ($n = 1, 2, 3, 4, 5 \dots$)
- (2) Filter media surface area standard: the flow rate to achieve the flux of 1 mL/min/cm^2 .

- P2: $\geq 100 \text{ kPa}$

NOTE 8: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

10.5.4 Turn on the injection pump which delivers the challenge solution prepared at ¶ 10.5.1.

10.5.5 Begin injection at rate to achieve 10^{6-8} particles/mL (calculated actual number diluted from the original GNP number or concentration).

NOTE 9: The challenge level shall be defined in terms of the concentration at the filter after diluting in the main flow. The challenge level shall be lower than the measurable maximum concentration (particles/mL) of the OPC.

10.5.6 Record downstream pressure reading.

10.5.7 Run test for 1 hour.

10.5.7.1 Record particle levels in at least ten minute intervals.