

SEMI C89-0116

TEST METHOD FOR PARTICLE REMOVAL PERFORMANCE OF LIQUID FILTER RATED BELOW 30 nm WITH INDUCTIVELY COUPLED PLASMA – MASS SPECTROSCOPY (ICP-MS)

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1 Purpose

1.1 This Document is to provide a standard of mono-dispersed gold nanoparticle (GNP) challenge test for liquid filter rated below 30 nm using inductively coupled plasma-mass spectroscopy (ICP-MS).

2 Scope

2.1 This Document covers a mono-dispersed GNP challenge test method for below 30 nm rated liquid filter.

2.2 This Document defines a test condition for mono-dispersed 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.
- 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 Fresh filter shall be used for this test procedure.

3.3 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.

3.4 Test parameters affect the outcome of the test described in this Standard, therefore test results are a qualitative rather than a quantitative of filter performance. This Standard recommends testing filters under the conditions optimized in this Standard and describe the conditions in test report.

3.5 Test shall be conducted as side by side comparison using more than one filter. Accordingly, the variability of the challenged particle in size is allowed in this test.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C79 — Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distributions Systems

SEMI C82 — Test Method for Particle Removal Performance of Liquid Filter Rated 20 to 50 nm with Liquid-Borne Particle Counter

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

5 Terminology

5.1 Abbreviations and Acronyms

- 5.1.1 *APD* — 2-amino-2-hydroxymethyl-1,3-propanediol
- 5.1.2 *DLS* — dynamic light scattering
- 5.1.3 *FM* — flow meter
- 5.1.4 *GNP* — gold nanoparticle
- 5.1.5 *ICP-MS* — inductively coupled plasma-mass spectroscopy
- 5.1.6 *LPM* — liter per minute (L/min)
- 5.1.7 *LRV* — log reduction value
- 5.1.8 *MSA* — mercaptosuccinic acid
- 5.1.9 *NIST* — National Institute of Standards and Technology
- 5.1.10 *OPC* — optical particle counter
- 5.1.11 *P* — pressure gauge
- 5.1.12 *PSL* — polystyrene latex
- 5.1.13 *SAXS* — small angle X-ray scattering
- 5.1.14 *T* — thermometer or thermocouple
- 5.1.15 *UPW* — ultrapure water
- 5.1.16 *V* — valve

5.2 Definitions

- 5.2.1 *background* — the gold concentration 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 (below 30 nm) with the water including ligand and GNP by calculating the difference of the number of challenged particle between upstream and downstream. The concentration of challenged GNP is measured with ICP-MS.

7 Apparatus

7.1 Test Device

- 7.1.1 For this Test Method, use the schematic shown in Figure 1.
- 7.1.2 The standard test device shall consist of a pre-filter, flow meters, a particle and ligand injection device, static mixer, 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 pre-filter 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 or thermocouple 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.

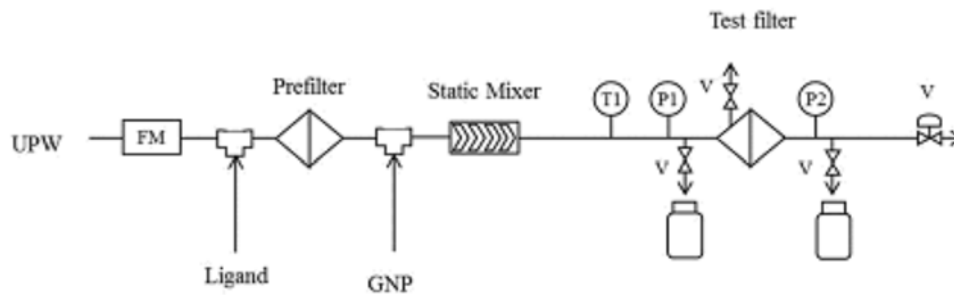


Figure 1
Schematic of Typical Test Setup (Example)

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 ICP-MS

- 7.4.1 Use an ICP-MS capable of detecting more than 0.1 ppb quantitative limit of gold.
- 7.4.2 Sample solution containing GNP shall be dissolved with aqua regia prior to injecting into ICP-MS.

8 Reagents and Materials

8.1 Ultrapure Water (UPW)

- 8.1.1 Use the supply UPW with the resistivity more than $17\text{M}\Omega\cdot\text{cm}$.
- 8.1.2 Temperature of the UPW shall be adjusted to $25 \pm 5^\circ\text{C}$.

NOTE 1: 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 mono-dispersed GNP certified by NIST or the mono-dispersed NIST-traceable GNP.
- 8.2.2 The mono-dispersed GNP with below 30 nm of particle size is used.

NOTE 2: In Appendix 2, the average particle size of NIST reference materials (RM) is described.

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 virgin liquid filter from rating below 30 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).

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

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

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 $>0.1\text{ mL/min/cm}^2$.
- P2: $\geq 100\text{ kPa}$

NOTE 3: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

10.2.3 Collect the test line water and measure gold concentration with ICP-MS.

10.2.4 Confirm the concentration becomes below detection limit, and record.

10.3 Filter Pretreatment

10.3.1 Pre-wetting 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 4: 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 Close the air-vent valve after venting air from the filter upstream.

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

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

Choose either standard below:

1. Filter size standard: n L/min at $25.4 \times n$ mm-sized (n inch-sized) filter, ($n=1,2,3,4,5, \dots$)
 2. Filter media surface area standard: the flow rate to achieve the flux of >0.1 mL/min/cm².
- P2: ≥ 100 kPa

NOTE 5: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

10.4.4 Collect the downstream of the filter and measure gold concentration with ICP-MS.

10.4.4.1 Record the concentration.

NOTE 6: If the gold concentration does not become below detection limit, 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 mono-dispersed 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. Preferably, the particle size in the solution also should be evaluated with the instrument such as DLS, and SAXS in case the particle aggregation/agglomeration might occur. The purpose of this measurement is to confirm that no existence of large aggregated/agglomerated particle in the challenge solution. Large particles may affect removal efficiency of the tested filter, which may cause overestimate the removal capability of the filter. If obviously larger particles than estimated challenge particle size are observed, the challenge test shall be terminated.

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

- FM

Choose either standard below:

1. Filter size standard: n L/min at $25.4 \times n$ mm-sized (n inch-sized) filter, ($n=1,2,3,4,5, \dots$)
 2. Filter media surface area standard: the flow rate to achieve the flux of >0.1 mL/min/cm².
- P2: ≥ 100 kPa

NOTE 7: A downstream pressure of at least 100 kPa has to be maintained in order to eliminate air in the system.

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

10.5.4 Begin injection at rate to achieve <100 ppb (calculated actual number diluted from the original GNP concentration).

NOTE 8: The challenge level shall be defined in terms of the concentration at the filter after diluting in the main flow. Record downstream pressure reading.

10.5.5 Run test for achieving below the order of 10^{10} pcs/cm² of the number of total challenged particle by tuning challenged GNP concentration and time. Table 1 summarizes examples of the testing condition.

Table 1 Examples of the Test Condition in Terms of Concentration Filter Surface Area, and Challenging Time

GNP size (nm)	GNP concentration (ppb)	GNP particles (pcs/ml)	Flow rate (L/min)	Filter Surface area (cm ²)	Time (min)	Accumulated challenge level (pcs/cm ²)	Membrane coverage area (cm ²)	Membrane Coverage rate (%)
20	10	7.0E+7	10	20,000	60	2.1E+9	131.9	0.7
10	5	2.9E+8	10	20,000	60	4.3E+9	67.2	0.3

10.5.5.1 Record particle levels in upstream and downstream each.

10.5.5.2 Monitor particle levels downstream of the filter.

10.5.6 Turn off injection pump.

10.5.7 Turn flow off at inlet valve.