

SEMI C77-0912 (Reapproved 0618)

TEST METHOD FOR DETERMINING THE COUNTING EFFICIENCY OF LIQUID-BORNE PARTICLE COUNTERS FOR WHICH THE MINIMUM DETECTABLE PARTICLE SIZE IS BETWEEN 30 nm AND 100 nm

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1 Purpose

1.1 This Document is to provide a test sequence for determining the counting efficiency of liquid-borne particle counters for which the minimum detectable particle size is between 30 nm and 100 nm.

2 Scope

2.1 This Document covers the counting efficiency test system for liquid-borne particle counters by using the quantified particle number concentration sample.

2.2 This Document defines the test conditions for the counting efficiency test of liquid-borne particle counters.

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

- Mass concentration of original polystyrene latex (PSL) particle suspension
- Factor for calculating the dilution ratio such as fluid, flow rate, pressure, volume, PSL particle number concentration, etc.
- Type of an equipment under test (EUT) liquid-borne particle counter
- Type of PSL particles
- Description of the test report

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 This Test Method is limited to testing the counting efficiency by measuring mono-dispersed PSL particles. However, actual particle size distribution is mostly poly-dispersed and the size distribution varies. So, there may be a certain level of difference between this test result and actual measurement data.

3.2 This Test Method is not in opposition to the concepts for the test method of the counting efficiency defined by ISO 21501-2. Rather, it may become a test method effective in the performance check of the reference instrument for which is requested by ISO 21501-2.

3.3 In this standard practice, in order to enable actual practice, the particle number concentration of test PSL particles suspension is obtained from a mean size and mass concentration which a manufacturer defines. More exact number concentration may be obtained by consideration of the size distribution, and precision mass measurement. At least, it has been experienced that the number concentration obtained from the mean size and mass concentration from a manufacturer is properly reasonable in case of the over 50 nm PSL particles.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI F63 — Guide for Ultrapure Water Used in Semiconductor Processing

SEMI F104 — Particle Test Method Guide for Evaluation of Components Used in Ultrapure Water and Liquid Chemical Distribution Systems

4.2 ISO Standards¹

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

ISO 21501-2 — Determination of Particle Size Distribution – Single Particle Light Interaction Methods – Part 2: Light Scattering 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 *CV* — coefficient of variation

5.1.2 *EUT* — equipment under test

NOTE 1: The meaning in this Document is a particle counter tested.

5.1.3 *FC* — flow controller

5.1.4 *1st (First) Sample* — primary dilution PSL particle suspension

5.1.5 *FM* — flow meter

5.1.6 *LPC* — liquid-borne particle counter

5.1.7 *PSL* — polystyrene latex

5.1.8 *UPW* — ultrapure water

5.2 Definitions

5.2.1 *background* — the number counts of LPC when supplying UPW.

5.2.2 *coincidence loss* — counting loss of particles which occurs if two or more particles exist simultaneously within a particle detection area or signal processing time.

5.2.3 *minimum detectable particle size* — smallest particle size that LPC can show as threshold.

NOTE 2: The below terms are defined in ISO 21501-2.

- size resolution
- counting efficiency
- false count rate
- minimum detectable particle size

6 Apparatus and Reagent Materials

6.1 Equipment Under Test (EUT)

6.1.1 EUT is LPC which uses a light scattering principle for the particle detection method.

6.1.2 The minimum detectable particle size of the EUT should be between 30 nm and 100 nm.

6.1.3 It is recommended that EUT is within the term of validity of the calibration certificate by the manufacturer.

6.2 UPW

6.2.1 Use the UPW with the resistivity more than 17 MΩ cm.

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

6.2.3 Refer to SEMI F63 for the other qualities of the UPW.

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6.2.4 Operating pressure downstream of the EUT should be >100 kPa (gauge pressure) during testing. The limit of this pressure value is not required when it is confirmed that FC and FM can operate normally.

6.3 Particle Injection Device

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

6.3.2 The accuracy of an injection flow rate and the pressure range of use shall be specified.

6.4 PSL Particle

6.4.1 Use the mono-disperse PSL particles, that is traceable to an international standard of length, and where the standard uncertainty of the mean particle size is equal to or less than $\pm 2.5\%$.

6.4.2 Since a width of size distribution of test PSL particles affects the uncertainty of measurement results (see § A1-4 in Appendix 1), the test PSL particles by which the standard deviation of particle size distribution is disclosed shall be used.

6.4.3 Main suspension material is UPW. Any materials, such as high concentration surfactant which may influence the size distribution of PSL particles should not be included.

6.5 Test Environment

6.5.1 The recommended cleanliness of the test environment is greater than ISO Class 7 defined in ISO 14644-1.

6.5.2 Temperature of the test environment shall be adjusted to $25 \pm 5^\circ\text{C}$.

7 Procedure

7.1 Test Circuit

7.1.1 For this Test Method, use the schematic shown in Figure 1.

7.1.2 The typical test system should consist of a pre-filter, flow controllers (FC) and meters (FM), particle injection device, EUT, a resistivity sensor, a thermometer and tubing connecting them. However, when the performance of UPW supplied to the test circuit has been clarified, pre-filter, resistivity sensor, thermometer, and pressure gauge are not necessarily required in this test system.

7.1.3 The pore size of a pre-filter or the specification of supplying UPW shall be smaller than the evaluated particle size. However, the quality of UPW may be unable to be controlled only by performance specification of a pre-filter. Checking by background test is important for the control of cleanliness factor of UPW. Also, the specification of a pre-filter, and its installation should be considered suitably by the result of the background test.

7.1.4 Use a flow meter of an allowable error to be less than 5% full scale and of appropriate range. More correct flow rate can be obtained by measuring the volume of the fluid by a graduated cylinder or measuring the mass of the fluid by a weighting scale, in the given time measured by a stopwatch. It is also the preferred approach to calibrate the flow meter by the above method.

7.1.5 Outline of the Procedure of Figure 1

7.1.5.1 UPW is supplied by a constant flow rate and the flow rate shall be controlled by the sum of the sample flow rate (U_2) of a particle counter and the bypass flow rate (U_3).

7.1.5.2 The pressure of UPW in the test system shall be within the operating pressure range of the EUT, and shall be below the discharge pressure of the injection pump.

7.1.5.3 The diluted PSL test particles by the procedure of Figure 2 are continuously injected into UPW with a constant flow rate by the injection pump.

7.1.6 For the dilution method of original PSL particle suspension, use the schematic shown in Figure 2.

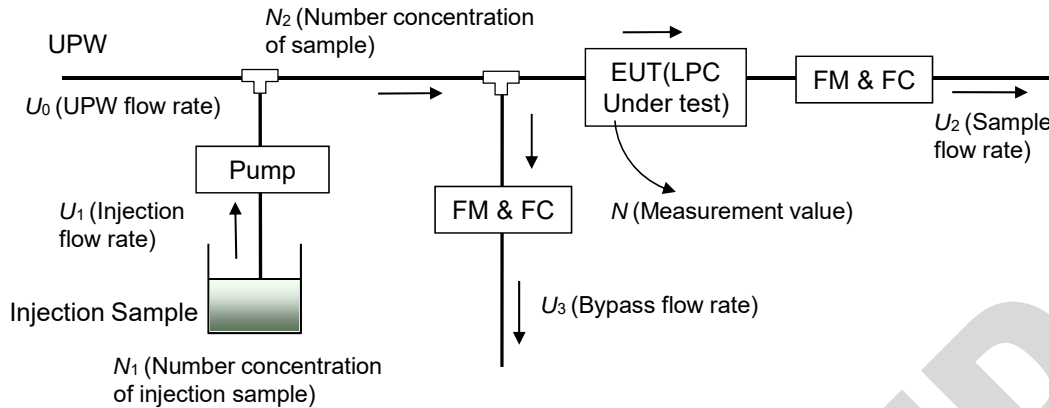


Figure 1
Schematic Test Setup

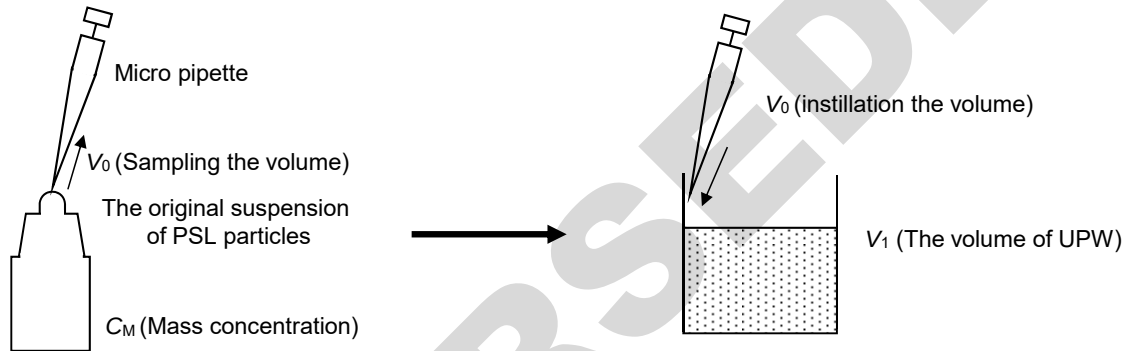


Figure 2
Schematic Procedure of the Dilution of the Original PSL Particle Suspension

7.2 Setting the Value of Parameter

7.2.1 The PSL particle number concentration (N_2) introduced to EUT shall be less than the maximum particle number concentration (N_L) of the EUT, since the concentration should not make the significant coincidence loss. Also if the number concentration is too low, the statistical error of measured value becomes large. Therefore, the concentration should be adjusted so that the measured value for 1 minute exceeds at least 1000.

7.2.2 Set the following each flow rate and volume. Accuracy of the each equipment shall be clarified.

7.2.2.1 Sampling volume of the original suspension of PSL particles (V_0).

7.2.2.2 Volume of the UPW for diluting the original suspension of PSL particles (V_1).

7.2.2.3 Injection flow rate of the diluted PSL particles (U_1).

7.2.2.4 Bypass UPW flow rate (U_3).

7.2.3 N_2 is defined by the following Equation 1, and N_0 means the number concentration of original suspension of PSL particles (see § 7.3) and U_2 means the rated flow rate of the particle counter. In fact, $U_0 + U_1$ can be measured by $U_2 + U_3$.

$$N_2 = N_0 \frac{V_0}{(V_0 + V_1)} \frac{U_1}{(U_0 + U_1)} \quad (1)$$

7.2.4 Example of Setting Each Parameter — In case that the mean size of PSL test particles is 61 nm, the mass concentration is 1% (the number concentration of the PSL particles corresponds to 7.8×10^{13} #/mL) and the maximum measurable number concentration of EUT is 24000 #/mL, following setting values are recommended.

- V_0 : 1 μ L
- V_1 : 1000 mL
- U_0 : 1000 mL/min
- U_1 : 50 μ L/min
- U_2 : 20 mL/min
- $N_2 = 7.8 \times 10^{13} \times (0.001 / [1000 + 0.001]) \times (0.05 / [1000 + 0.05])$
= 3900 #/mL

NOTE 3: When the EUT has a bypass flow system in its inside, it should be taken care for the actual sample flow rate setting.

7.3 Preparing 1st Test Sample (Primary Dilution PSL Particle Suspension)

7.3.1 The number concentration of original PSL suspension (N_0) is found from the specified mass concentration (C_M) of a PSL particle original suspension by the following Equation 2. In case of PSL particles, the shape of particle can be assumed spherical and the number-mean-size (D_0) can be used as typical particle size.

$$N_0 = \frac{C_M \rho_0}{V_p \rho_p} \quad (2)$$

$$\therefore V_p = \frac{\pi D_0^3}{6} \quad (3)$$

where, V_p is the volume of the PSL particle, ρ_p is the density of the particle and ρ_0 is the density of original PSL suspension. Actual ρ_0 can be treated as almost same as the density of UPW (see Equation A1-14 in Appendix 1).

NOTE 4: C_M is a ratio of the mass of the containing total solid to the mass of original particle solution.

7.3.2 The volume of V_0 is determined by the sampling which used micro-pipette.

NOTE 5: The accuracy of micro-pipette should be clarified.

NOTE 6: Contamination of micro-pipette should be fully eliminated.

7.3.3 The sampled original suspension of PSL particle is dropped into UPW of known volume (V_1).

NOTE 7: Cleanliness of the UPW and the pod should be enough.

7.3.4 The number concentration of 1st test sample means the number concentration of injection sample (N_1). N_1 is shown as following Equation 4.

$$N_1 = N_0 \frac{V_0}{V_0 + V_1} \quad (4)$$

7.4 Test-Line Background

7.4.1 This test is required before injecting test PSL particles (see Figure 1).

7.4.2 Set up the EUT into the test circuit.

7.4.3 U_1 , U_2 and U_3 shall be set to the same conditions as the test.

7.4.3.1 The injection sample shall be UPW.

7.4.3.2 The supply pressure shall be less than the specification of EUT.

7.4.4 The measurement shall be continued for more than 10 minutes, in the state where measured value is stabilized,

7.4.4.1 Record the measured number of particles in at least 1 minute intervals.