

SEMI C79-0819

GUIDE TO EVALUATE THE EFFICACY OF SUB-15 nm FILTERS USED IN ULTRAPURE WATER (UPW) DISTRIBUTION SYSTEMS

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NOTICE: This Document was completely rewritten in 2019.

1 Purpose

- 1.1 This Guide contains recommendations for evaluating the efficacy of filters used in ultrapure water (UPW) fluid streams. The filters control particles to prevent particle contamination on wafers and other critical surfaces.
- 1.2 Due to the lack of adequate on-line particle detection metrology at the particle sizes addressed by this Guide, off-line filter performance testing can be used to validate filter retention efficiency. Off-line filter performance testing helps mitigate the risks associated with particle control in flowing UPW streams in contact with silicon wafers.
- 1.3 This Guide contains recommendations for evaluating filters used in UPW fluid streams.
- 1.4 This Guide is not a standard. The procedures described in this Guide challenge filters with high-particle concentrations because current particle detection technology cannot detect low particle concentrations at sub-15 nm (representative of the real world).

2 Scope

- 2.1 This Guide applies to UPW system final filters as well as to other UPW distribution filters intended for use with semiconductor manufacturing tools and ancillary equipment.
 - 2.1.1 This Guide is recommended for additional applications, such as hot ultrapure water (HUPW) and dilute hydrophilic high-purity chemicals (requiring sub-15 nm particle control).
- 2.2 This Guide applies to advanced semiconductor facilities requiring control of the UPW particles from 5 to 15 nm. While filters may provide effective removal of sub-15 nm particles, the most penetrating particle (MPP) size may be significantly larger. The scope of this Guide includes measurement of the MPP size in the range up to 30 nm.
 - 2.2.1 This Guide may also be used for large particles not covered by existing particle-detection metrology.
- 2.3 The following three criteria for evaluating filter performance are considered in this Guide:
 - 2.3.1 A particle challenge test assesses the ability of the filter to retain influent particles. The filter is tested under single-pass water filtration or with a challenge particle solution (no recirculation applied).
 - 2.3.2 An initial filter rinse assesses the potential contribution of particles by the filter.
 - 2.3.3 Repeating the performance test throughout the operating lifetime of the filter addresses filter performance stability. Different representative filters from the same group of filters are tested during filter life time in order to confirm the frequency of the filter change out.

NOTE 1: Major defects in the filter are detected using an integrity test under conditions specified by the filter supplier.

- 2.4 This Guide describes procedures for measuring filter particle retention while using UPW as the test media. UPW parameters, for the intents and purposes of this Guide, are defined in § 7.6.
- 2.5 This Guide uses commercially available colloidal silica with particle sizes of 5 to 30 nm. Colloidal silica is a superior challenge material representative of the conditions of the UPW system operation. It is commercially available (and inexpensive) in the 5 to 30 nm size range (Reference Grant DC, 2011). “The effect of particle composition on filter removal of sub-30 nm particles from UPW” presented at the Executive Forum at ULTRAPURE WATER Micro 2011, Portland, OR.
- 2.6 The filter-performance evaluation procedure described in this Guide uses a combination of grab-sample lab analysis and in situ liquid particle measurement using an aerosol particle detection technique.

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 Guide maximizes particle measurement sensitivity and standardizes the testing environment. However, actual performance under normal operating conditions may vary.

3.2 Filters tested under the conditions specified in this Guide may vary in their performance from the filters used in the actual UPW system. Consistency of the filter manufacturing may also affect test results therefore testing more than one filter and conducting a statistical analysis should be considered.

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

3.4 The challenge-test conditions use a particle concentration significantly higher than the concentration typically expected in the UPW feed into the final filters. Therefore, the performance of the filters under typical UPW conditions is expected to be better than their performance in the test. This Guide should be used by the end user to assess filter performance; it should not be used by filter suppliers to rate filter performance.

3.5 This Guide can be used to test smaller filters than those typically used in production-scaled UPW applications. For valid test outcomes, small filters should be verified by the filter supplier to have the same performance characteristics as their larger counterparts (such as ultra-filters [UF] or greater numbers of single typical cartridge filter elements [0.25 m size] connected). For the validity of the results, the flow rate through the tested filter should be scaled to the actual operating conditions.

3.5.1 Filter material cleanliness and potential leaching of dissolved contaminants are also addressed by this Guide.

3.6 The accuracy of the data generated by the challenge test described in this Guide is limited to the accuracy of the analytical techniques used.

3.7 The experimental work conducted in preparation for this Guide suggested that using a high-challenge particle concentration significantly affects filter retention efficiency (see Appendix 5). The required efficiency of the filtration depends on the end user's process needs and will be finally determined by the end user.

3.8 The required efficiency for particle removal is to be confirmed by the end user based on the process need.

3.9 Filter operational conditions used in the test (such as flux, differential pressure, specific surface area, etc.) are limited to the information provided by the filter supplier. The end user should compare the conditions of the test to those used in the actual application to ensure that the results of the tests are representative.

3.10 Tolerances in the figures used in the test protocol (such as flow rate, concentration, etc.) are assumed to be $\pm 10\%$ unless otherwise stated.

3.11 Minimum size or number of the tested filters should provide sufficient flow rate to adequately operate the metrology used in the test. To provide the minimum turbulence required for consistent performance, a Reynolds Number $> 5,000$ is required in all main process piping.

3.12 Particle loading rate and concentration are fixed in the specified test conditions to prevent excessive loading in units of particle equivalent surface coverage (monolayers) of colloidal silica on the filter surface.

3.13 The Guide is limited to the conditions of ambient temperature of UPW. Performance of the filters under HUPW conditions has been excluded from this Guide due to limited data available. The HUPW filter testing is expected to be included in a future revision of this Guide.

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 Materials and Components Used in Ultrapure Water and Liquid Chemical Distribution Systems

SEMI F61 — Guide for Ultrapure Water System Used in Semiconductor Processing

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

5 Units

- 5.1 Parts per million (ppm) is equivalent to $\mu\text{g/mL}$ or mg/L , where 1 L approximately equals 1 kg.
- 5.2 Parts per billion (ppb) is equivalent to ng/mL or $\mu\text{g/L}$, where 1 L approximately equals 1 kg.
- 5.3 Parts per trillion (ppt) is equivalent to pg/mL or ng/L , where 1 L approximately equals 1 kg.
- 5.4 Micrometer is a unit of length equal to one millionth of a meter, or one thousandth of a millimeter.

6 Terminology

6.1 Abbreviations and Acronyms

6.1.1 General terms and acronyms used in this Standard are listed below and may be defined in SEMI F61.

- 6.1.1.1 *DMA* — differential mobility analyzer
- 6.1.1.2 *HUPW* — hot ultrapure water
- 6.1.1.3 *ICP-MS* — inductively coupled plasma mass spectrometry
- 6.1.1.4 *ITRS* — International Technology Roadmap for Semiconductors
- 6.1.1.5 *NCPC* — nebulization with condensation particle counter
- 6.1.1.6 *NRM* — nonvolatile residue monitor
- 6.1.1.7 *OPC* — optical particle counter
- 6.1.1.8 *PSDA* — particle size distribution analyzer
- 6.1.1.9 *PSL* — polystyrene latex
- 6.1.1.10 *PSD* — particle size distribution
- 6.1.1.11 *TOC* — total organic/oxidizable carbon
- 6.1.1.12 *UPW* — ultrapure water

6.2 Definitions

6.2.1 *background* — the average particle and other contaminant concentrations in the test system reported by OPC, PSDA, NRM, dissolved silica and total silica analyses. Background is reported when UPW flows through the test skid and spool piece (after rinsing the test skid components and spool piece to a steady-state of background contamination). Background includes contributions from the UPW and test skid components.

6.2.2 *test skid* (see Figure 1) — the system providing filter-evaluation test analysis. The test skid includes piping, filter housing, filter elements, flow meters, pressure gauges, valves, regulators, sample ports, etc.

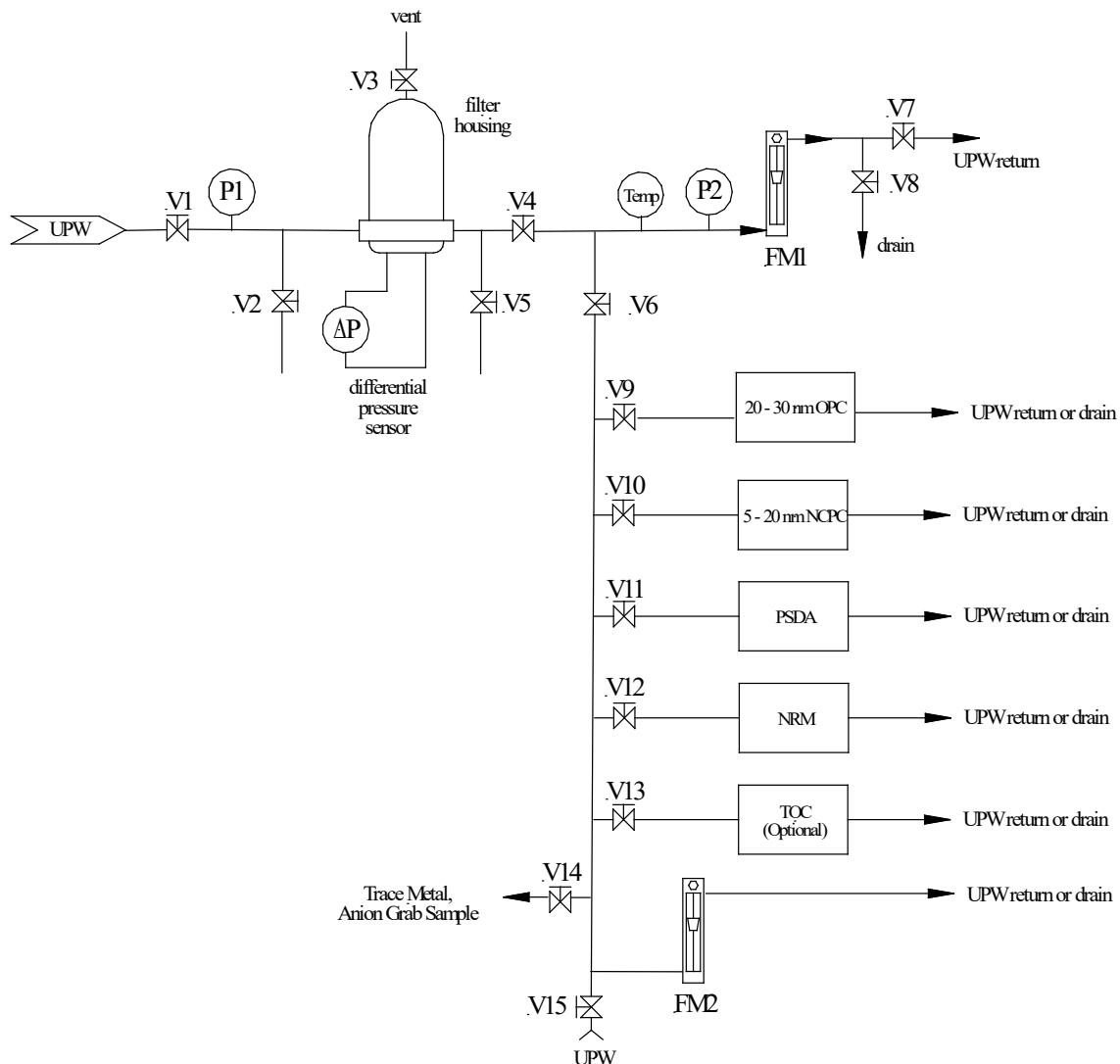


Figure 1
General Test Schematic Diagram

NOTE 2: In Figures 1 through 4 the differential mobility analyzer (DMA) (the key element of the LSNP) tool is used as an example of a particle size distribution analyzer (PSDA). DMA should be viewed as a PSDA when mentioned in the text throughout the Document. PSDA may have different types and configurations. The diagram represents an example, where LSNP is used, which provides on-line measurement. However, different types of PSDA may require grab sample and on-line installation would not be needed.

NOTE 3: Collecting accurate total organic/oxidizable carbon (TOC) flush data may require dedicated TOC instrumentation upstream and downstream of the filter adding cost and complexity to the test. Switching between upstream and downstream points is unreliable and is not recommended. Measuring TOC during the rinse test is optional and should only be used for reference.

7 Test Skid Configuration

7.1 The test skid should be enclosed in an ISO Class 8 (per current revision of ISO 14644, roughly equivalent to FED STD 209E Class 100,000), or better, environment. Procedures necessary to maintain ISO Class 8, or better, when handling any part of the test system, or during testing, should be followed. Deviation from this requirement may be

approved by the end user, while maintaining the intent of the clean environment. If more exacting testing is required, end users may need to follow more stringent testing requirements.

7.2 Ultrapure, low-particle-generating, low shedding piping systems should be used in all wetted flow paths from the UPW source to the filtration test skid to ensure that the requirements of § 7.6 are maintained. The recommendations within SEMI E49 should be considered when designing and assembling the system.

7.3 The test skid should be designed and built according to the requirements listed in Appendix 1.

7.4 The method of injection is flexible provided a clean environment can be ensured. Reversing the flow in the injection port is necessary to prevent stagnation and contamination.

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

7.6 The UPW should be in compliance with the following minimum characteristics:

7.6.1 Minimum operating pressure downstream from the test skid should be 30 psig (138 kPa) during testing.

7.6.2 Acceptable temperature for testing can range from 18°C to 28°C with a maximum of $\pm 1^\circ\text{C}$ variation over duration of the test ($77 \pm 9^\circ\text{F}$). (Concerns: viscosity, gas, diffusion—need to possibly tighten the range.)

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

7.6.4 TOC <5 ppb (sensor is temperature compensated).

7.6.5 NVR <200 ppt.

7.6.6 The maximum recommended particle-concentration background level is 0.5 particle/mL ($\geq 0.05 \mu\text{m}$).

7.6.7 It is recommended that incoming water be degasified to minimize the risk of bubble formation that might interfere with particle measuring instrumentation. Degassing the incoming water will also aid in ensuring full wetting of the filter during installation and rinse.

7.7 Before beginning the test, filter components should be prepared, installed, and pre-wetted in accordance with the filter supplier recommendations.

7.8 Ensure that the instrumentation (flow meters and pressure gauges or transducers) is calibrated in accordance with the manufacturers' procedures and frequency.

7.9 The components under test and the plumbing between the components under test should be oriented to limit bubble entrapment because bubbles cause metrology errors. The filters should be positioned vertically, and the test skid should be vented to prevent gas accumulation and bubble formation.

7.10 A data-acquisition system is required to monitor filter performance. The data acquisition system should be able to capture particle data at least once per minute.

7.10.1 To test the background level of the test skid, configure the test skid in accordance with Figure 2 and insert a spool piece or empty filter housing as needed (the background test provides baseline measurement data needed for the initial rinse-up reference). When testing cartridge filters, it is recommended to use an empty filter housing instead of a spool piece.

7.11 For rinse testing, configure the test stand in accordance with Figure 3 and insert a test filter component or test filter assembly.

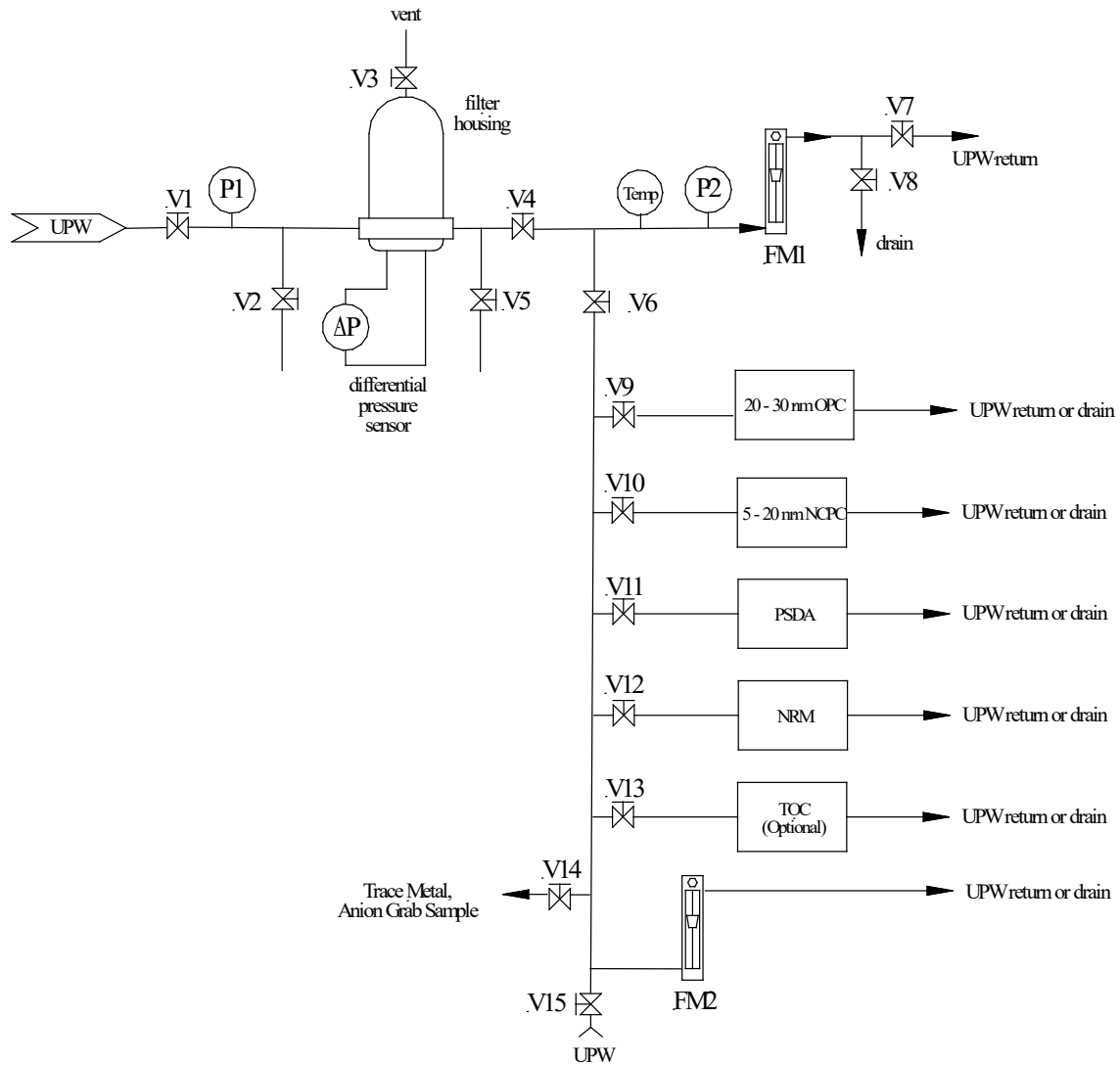


Figure 2
Background Test Configuration

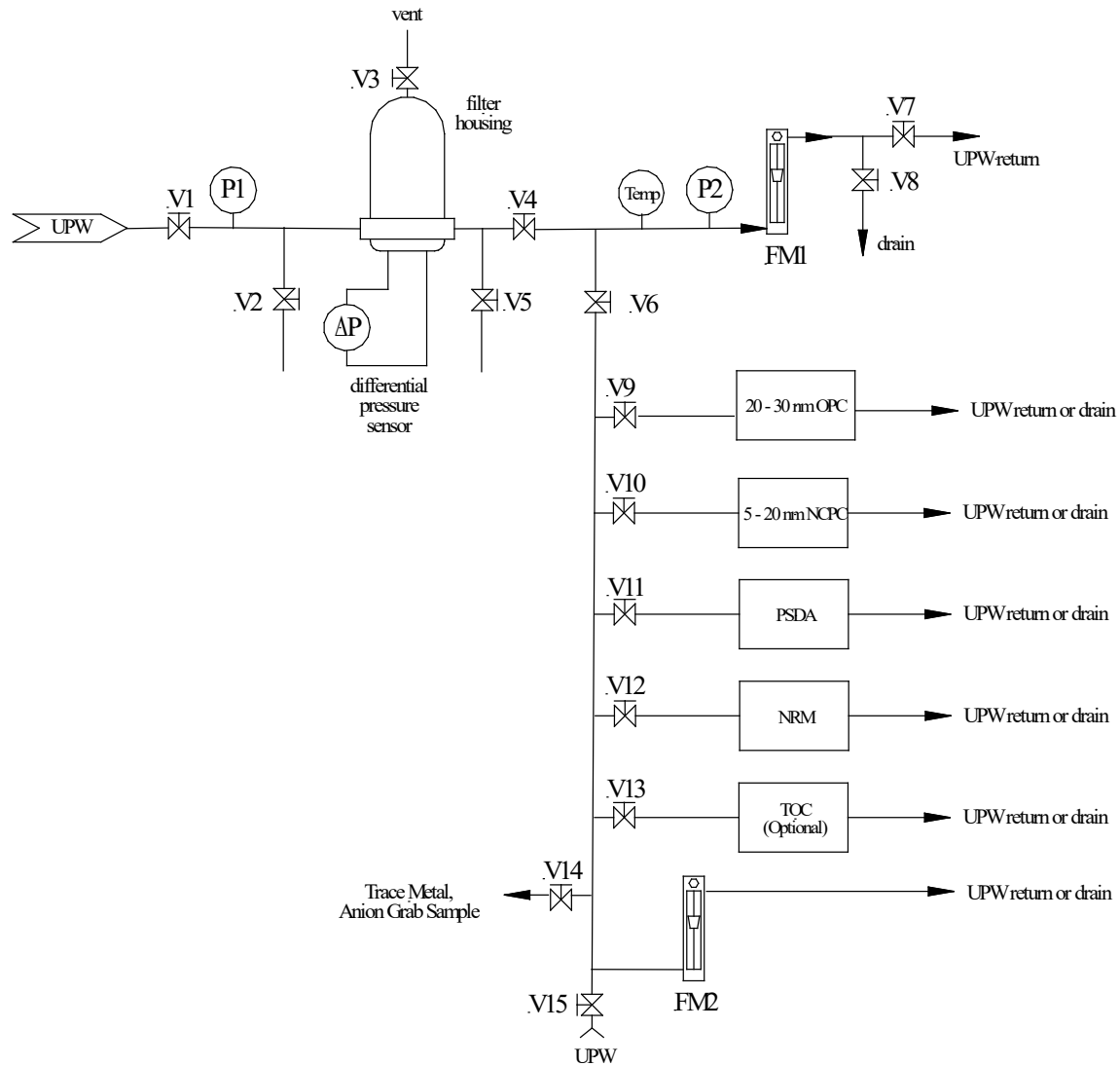


Figure 3
Rinse-Up Test Configuration

7.12 For the particle challenge test, configure the system in accordance with the schematic diagram shown in Figure 4.

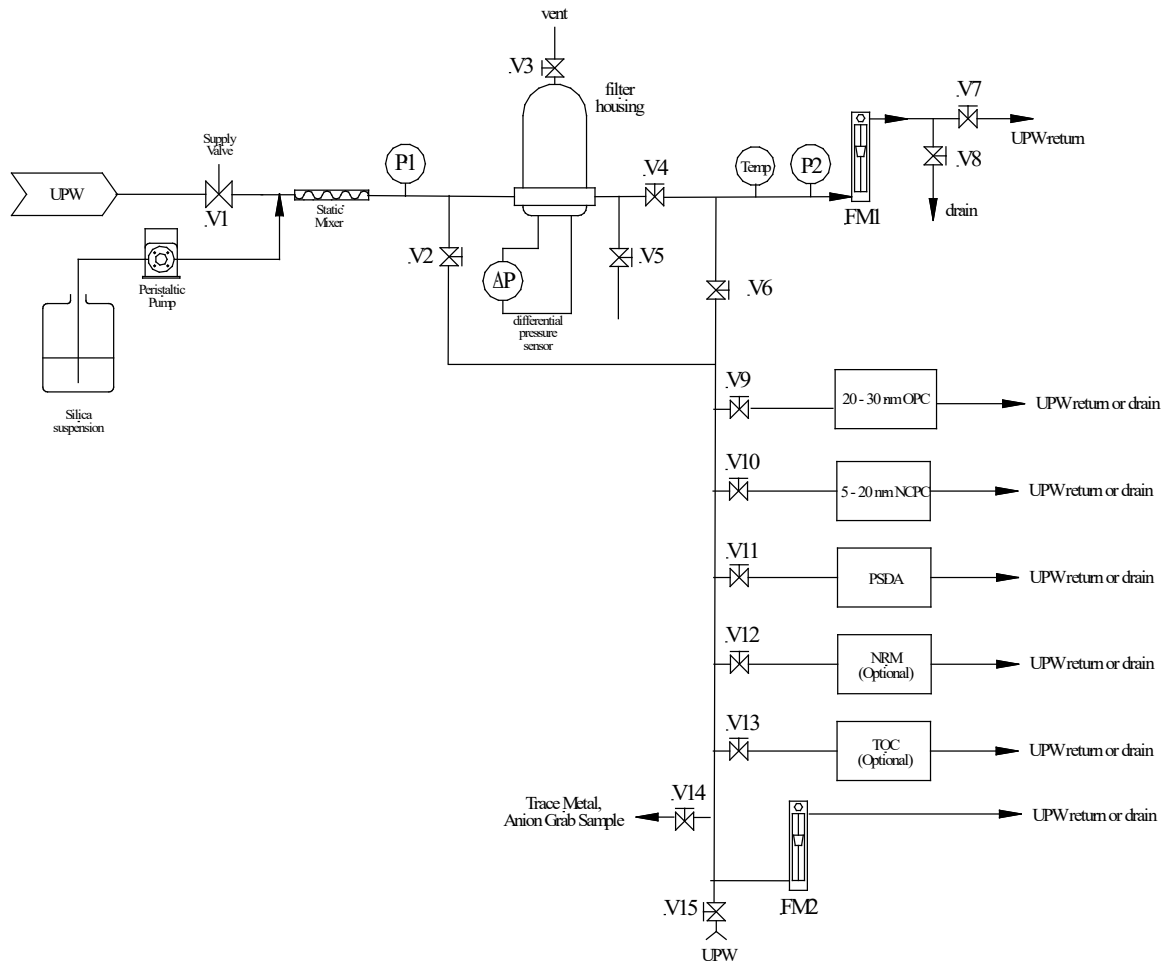


Figure 4
Particle Challenge Test

8 Special Recommendations

8.1 Material purity leach data (based on SEMI F40 method) should be assessed for compatibility of the filters with high purity application.

8.2 A minimum of three filter components or component assemblies should be tested to assess variability. Component handling and packaging may impact particle test results. When testing, component handling and packaging by the filter manufacturer should be in accordance with the typical manufacturing processes.

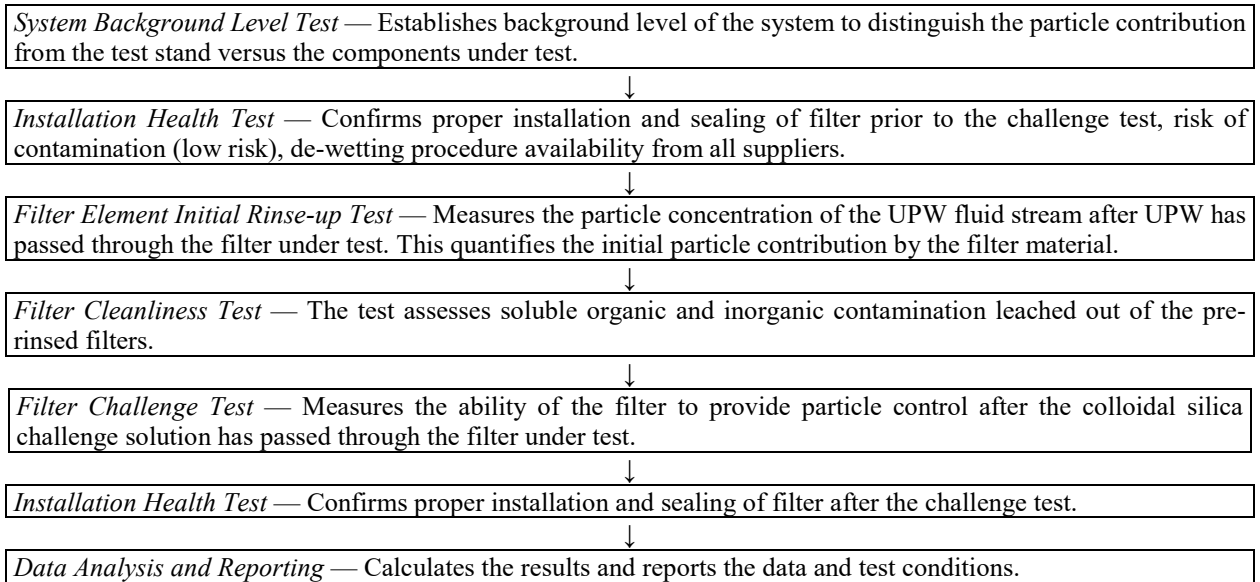
9 Procedures

9.1 General Procedures

9.1.1 Throughout this Document, the instructions referring to the starting and stopping of UPW flow should be carried out by using valves located at 'From UPW Supply' and 'To return or drain,' as shown in Figures 1 through 4.

9.1.2 The steps referring to monitoring and adjusting the flow should be carried out using the flow meter and the bypass flow meter shown in Figures 1 through 4. The flow through the flow meters should remain constant, as specified by the manufacturer.

9.1.3 This Guide uses the following progression for analyses of a filter's capability of providing particle control in UPW applications.



9.2 Test Conditions

9.2.1 Given the possibility that different types, sizes, and configurations of the filters may be tested, the filter parameters, test conditions, and results should be reported in Tables 1 and 2 for effective comparison.

Table 1 Test Conditions Report

<i>Parameter/Conditions</i>	<i>Data</i>
Cleanliness of the skid materials (per SEMI F57 or other) – in compliance (Yes/No)	
Challenge particles (supplier, size mean, size weighting (number or volume), lot number)	
Volume of the filter housing/dummy module (between isolation valves) Note: Useful for extraction test and dilution rates.	
O-ring/gasket type	
Housing material of construction	
Filter part number and serial number	
Flux per unit area	
Surface area	
Type of the tested filter (cross flow/dead end, etc.)	
Filter material of construction (membrane, support, core, cage, caps, etc.)	
Provide URL to the filter data sheet prepared by the manufacturer	

Table 2 Test Results

Integrity Test Conditions and Results					
Test pressure, allowable diffusive flow, success criteria – pressure decay					
Parameters	Test Duration (template – add more columns as needed – the timelines may vary for the different steps in the test procedure)				
	0.5 hr	1 hr	1.5 hr	2 hrs	2.5 hr
<i>Feed and Background Characteristics</i>					
Feed Flow Rate 1 (L/min)					
Feed Temperature, C					
Feed Dissolved Oxygen, ppb (if DO controlled)					
Feed dissolved and total Silica, ppb concentration					
Feed NVR, ppb					
Feed Resistivity, MΩ·cm					
Feed particles level, #/L, at >0.05 μ (if OPC available)					
Feed Pressure (psi)					
Product pressure (psi)					
<i>Initial Rinse</i>					
Feed Pressure (psi)					
Product pressure(psi)					
Flow Rate (L/min)					
Product dissolved and total Silica, ppb concentration					
Feed Particles by aerosol particle detection (both concentration and size distribution)					
Product Particles by aerosol particle detection (both concentration and size distribution)					
Product NVR, ppb					
Product particles level, #/L, at >0.1 μ					
<i>Particle Challenge Test</i>					
Feed Particles by aerosol particle detection (both concentration and size distribution)					
Product Particles by aerosol particle detection (both concentration and size distribution)					
Feed dissolved and total Silica, ppb concentration					
Product dissolved and total Silica, ppb concentration					

9.3 Particle Sizing Calibration (refer to Appendix 4 for reference)

9.4 Calibrate the PSDA used for the test with a NIST traceable standard such as Reference Material 8011, gold nanoparticles, nominal 10 nm diameter or similar. Due to limited availability and high cost of internationally

recognized reference materials (RMs), conducting this calibration for each test using RMs is not viable. To address this limitation, the particle counting instrumentation used in for this standard test can be made traceable using available RMs at or close to the sizes of interest and correlating to a stable silica particle (10, 20 and/or 30 nm). This correlated silica challenge solution can subsequently be used to validate the calibration of the PSDA.

9.4.1 At this time, the smallest RMs available are 10 and 30 nm gold (NIST RM 8011 and NIST RM 8012) and 20 nm silica (ERM FD100). These three RMs combined are considered suitable for establishing traceability of the particle sizing instrumentation using in this guideline.

9.4.2 Start the injection of the traceable standard and collect sizing data for a minimum of 10 minutes.

9.4.3 Determine the number-weighted mean for the reference material.

9.4.4 Determine the difference between the number-weighted mean measured by the PSDA and the transmission electron microscopy (TEM) particle size reported for the reference material. Use this number as the sizing correction factor applied to all the silica PSDA measurements.

9.4.5 The calibration process is considered successful if the difference between the mean silica measured by the PSDA and NIST reference standard mean value is not larger than $\pm 30\%$ of the NIST specified value (30% is chosen based on typical variability of the NIST reported results).

9.5 Background Test

9.5.1 Background testing is important before every test in a test series, unless testing multiple identical filters. Example: Performing a rinse test on four individual filters when it is not necessary to re-establish the system background between each of the filter samples, but it is necessary to achieve a stable level of the online instrument readings within 10% of the initial background.

NOTE 4: In case of high background level of contamination, similar test may need to be done for the housing. Depending on the size and configuration of the filter housing, the housing test may be a part of this test or done separately using similar conditions described in this Guide.

9.6 Configure the test skid in accordance with Figure 2. Install spool piece/dummy filter/empty filter housing at the location of the filter component under test.

9.7 All instruments and analytical techniques included in the experimental work should be used for the background test. The background should be tested with the same technique used for the performance analysis, as well as using OPC, TOC, NRM, NCPC and grab-sample lab analysis.

9.8 Using the following instructions, clean the system by rinsing with UPW to a system background contamination level as defined in § 7.6.

9.8.1 When there is no flow through the filter housing portion of the test, it is important to maintain UPW flow through the instrument manifold. This achieved by closing V6 and opening V15.

9.8.2 Start the UPW flow through flow meter FM1 only and adjust the flow rate to 10 LPM or the rinse flow rate established by the test requestor. Maintain this flow rate for a minimum of 5 minutes in order to rinse the test stand and eliminate air bubbles. Vent the system appropriately. Flow rate may be increased as needed to clear bubbles from the system.

9.8.3 Once housing or spool is adequately vented and free of bubbles, introduce flow to the instrument manifold by opening V6 and closing V15.

9.8.4 Adjust FM1 and FM2 as needed to establish the rinse flow rate and instrument manifold pressure. Flow rate through the instruments should be accounted for in determining the total flow rate through the filter housing. Operating pressure at P2 should be 30 ± 5 psig (207 ± 34 kPa), unless something different is specified by the metrology manufacturers.

9.9 Start the OPC, NRM, NCPC, TOC, and PSDA to determine the background particle, NVR, and TOC concentration. Set the counting interval of the OPC at 15 minutes. The maximum recommended system background level is 0.5 particle/mL ($\geq 0.05 \mu\text{m}$) as measured by the OPC. Reaching stable background within the level of impurity at or near the noise level of the online instruments used is critical for successful analysis of the cleanliness of the filter.