

SEMI C79-0113

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

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1 Purpose

- 1.1 This Document is a recommended guide to evaluate the efficacy of filter elements used in ultrapure water (UPW) fluid streams, providing particle control as risk mitigation for particle contamination on the wafer.
- 1.2 Due to the lack of adequate on-line particle detection metrology at the particle sizes addressed by this Guide, however off-line filter performance testing can be used to validate filter retention efficiency. Off-line filter performance testing will help mitigate the risks related to particle control in flowing UPW streams that make contact with silicon wafers.
- 1.3 This Document provides a guide to the test method to evaluate filter elements used in UPW fluid streams.
- 1.4 This Document is a guide rather than a standard. Additional data is necessary to correlate the results tested in the procedure using high challenge particle concentrations with the actual filter's performance to evaluate particle behavior under much lower particle concentrations in real world UPW. That is to say, in the method described in this Guide, filters are challenged at high particle concentrations not representative of real-world conditions because current particle-detection metrology cannot detect low particle concentrations.

2 Scope

- 2.1 This Guide applies to UPW-system final filters and other UPW distribution filters intended for use in semiconductor manufacturing tools and ancillary equipment.
 - 2.2 This Guide applies to advanced semiconductor facilities requiring control of the UPW particles from 5 to 15 nm. The guide may also be used for large particles not covered by existing particle-detection metrology.
 - 2.3 Four aspects in evaluating filter performance are considered in this Guide:
 - 2.3.1 Ability of the filter to retain influent particles is assessed with a particle challenge test. The filter is tested under single pass filtration of water or challenge particle solution—with no recirculation applied.
 - 2.3.2 Possible contribution of particles by the filter is evaluated during initial rinse of the filter.
 - 2.3.3 Filter performance stability is addressed by repeating the performance test throughout the operating lifetime of the filter. 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 specified by the filter supplier.
- 2.3.4 Initial filter cleanliness.
 - 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, is defined in § 7.6.
 - 2.5 This Guide uses commercially available colloidal silica with a mean particle size of 5 to 15 nm. Using polystyrene latex spheres (PSL) as a filter challenge does not work at the 5 to 15 nm size range because the PSL size distribution is too broad and using white spherical plastic beads is an unrealistic challenge for filter testing. Colloidal silica is a superior challenge material because it is commercially available (and inexpensive) in the 5 to 15 nm size range. Colloidal silica has a relatively narrow size distribution and has lower interaction with filter material; therefore it is primarily removed by a filter's sieving mechanism.
 - 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 specifies challenge test conditions that will enhance the particle measurement sensitivity of the test and standardize 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 A large number of parameters affect the outcome of the test described in this Guide therefore it is challenging to maintain perfectly consistent conditions from one test to another. The test results are a qualitative rather than a quantitative assessment of filter performance.

3.5 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 Document should not be used for establishing the rating of a filter by suppliers, but rather for the end user assessment of the filter performance.

3.6 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 together. For the validity of the results, the flow rate through the tested filter should be scaled to the actual operating conditions.

3.6.1 Material purity and potential leaching of dissolved contaminants are not addressed by this Guide. 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.

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

3.8 The experimental work conducted as part of the preparation of this Document suggested significant effect of the challenge particle concentration to the filter retention efficiency (see Appendix 5). As a result, this Guide uses a minimum filtration-efficiency requirement of 50% as a reference only, assuming that filters, which do not provide 50% particle removal pose a higher risk of not removing particles at the 5 to 15 nm size under normal conditions of the UPW system. The required efficiency of the filtration depends on the end user's process needs and will be finally determined by the end user.

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

3.10 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.

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

3.12 Minimum size or number of the tested filters should provide sufficient flow rate to adequately operate 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.

3.13 Particle loading rate and concentration are fixed in the specified test conditions to prevent excessive monolayers of colloidal silica from developing on the filter surface. The end user should consider whether the flux used for the test represents the actual conditions of the filter operation in the full-scale application.

3.14 The guide is limited to the conditions of ambient temperature of UPW. Performance of the filters under hot UPW (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 Document.

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 UPW

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 *NRM* — nonvolatile residue monitor

6.1.1.6 *OPC* — optical particle counter

6.1.1.7 *PSDA* — particle size distribution analyzer

6.1.1.8 *PSL* — polystyrene latex

6.1.1.9 *TOC* — total organic/oxidizable carbon

6.1.1.10 *UPW* — ultra pure 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.

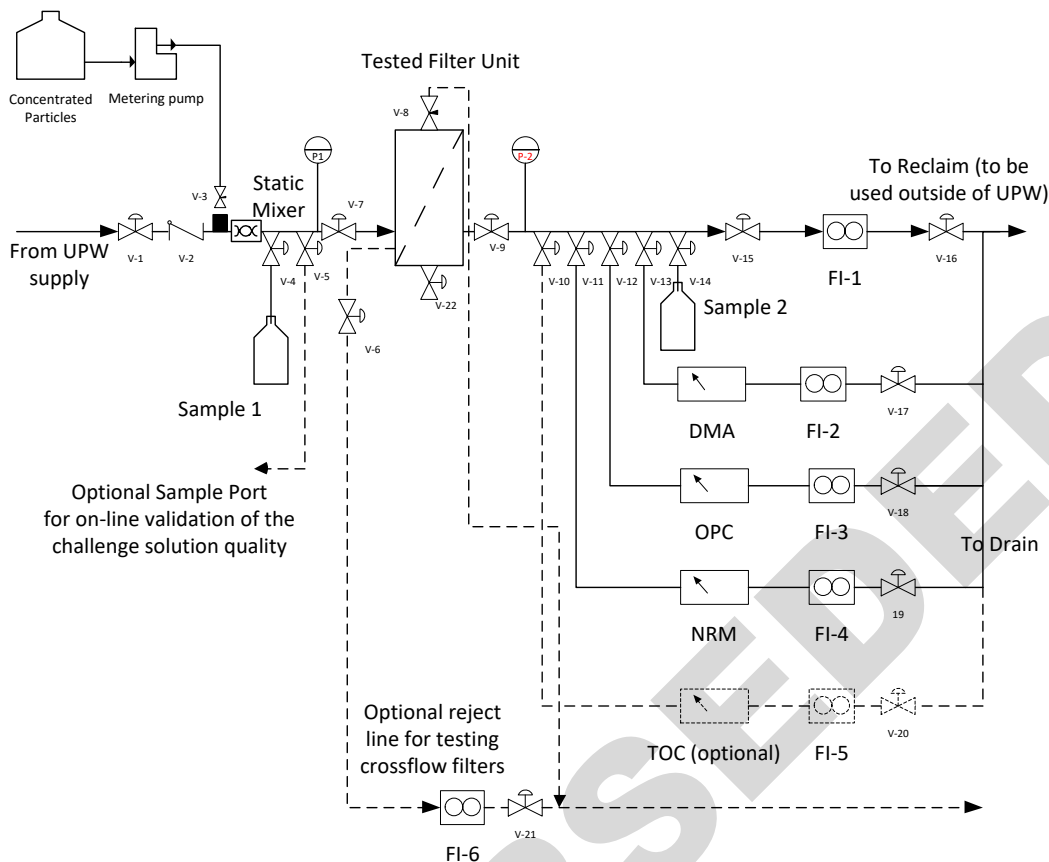


Figure 1
General Test Schematic Diagram

NOTE 2: In Figures 1 through 4 the DMA (the key element of the LSNP) tool is used as an example of a 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.

7 Test Skid Configuration

7.1 The test skid should be enclosed in an ISO Class 7 (per current revision of ISO 14644, roughly equivalent to FED STD 209E Class 10,000), or better, environment. Procedures necessary to maintain ISO Class 7, 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 requirements:

7.6.1 Minimum operating pressure downstream from the test skid should be 20 psig (138 kPa) during testing.

7.6.2 Temperature of $23 \pm 5^{\circ}\text{C}$ ($77 \pm 9^{\circ}\text{F}$).

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

7.6.4 Total organic/oxidizable carbon (TOC) < 5 ppb.

7.6.5 Total Silica < 0.5 ppb.

7.6.6 Dissolved Silica < 0.1 ppb.

7.6.7 NVR < 0.1 ppb.

7.6.8 The maximum recommended particle-concentration background level is 0.05 particle/mL ($\geq 0.1 \mu\text{m}$).

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.

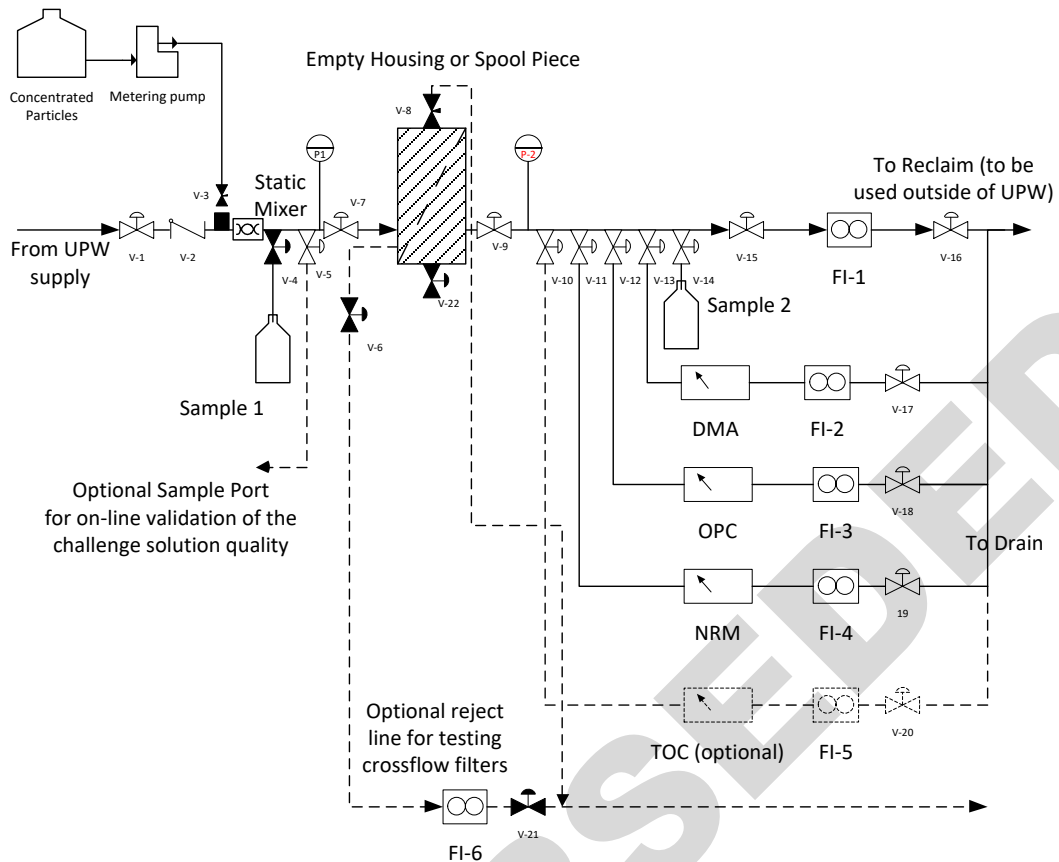


Figure 2
Background Test Configuration

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

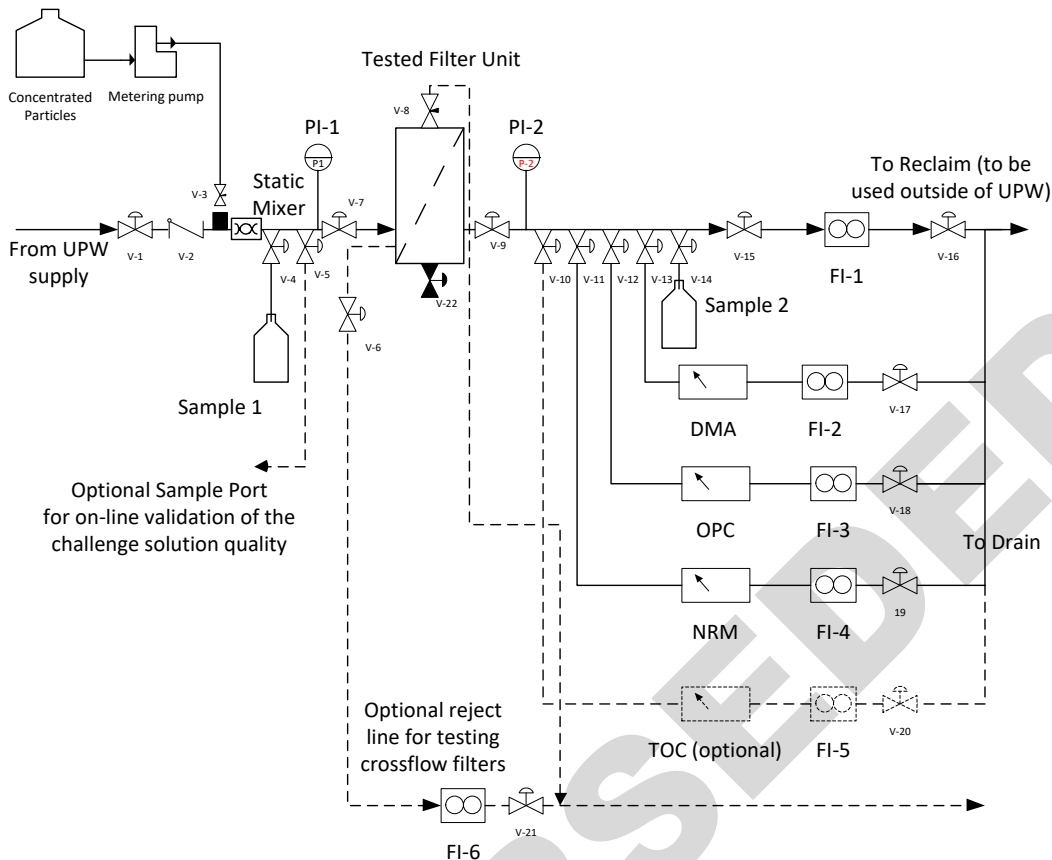


Figure 4
Particle Challenge Test

8 Recommendations

- 8.1 A representative filter sample of the population of filters under interest should be selected for testing.
- 8.2 Provide material purity leach data (SEMI F57) prior to the test to demonstrate compatibility of the system with high purity application. SEMI F57 covers all materials, but filters and the filter housings.
- 8.3 A minimum of four filter components or component assemblies should be tested to assess variability.
- 8.4 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 Reclaim/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.

System Background Level Test — establishes background level of the system to distinguish the particle contribution from the test stand versus the components under test.



Integrity Test — confirms filter integrity prior to the challenge test.



Filter Element Initial Rinse-up Test — measures the particle concentration of the UPW fluid stream after UPW has passed through the filter under test. This quantifies the initial particle contribution by the filter material.



Filter Cleanliness Test — the test assesses soluble organic and inorganic contamination leached out of the pre-rinsed filters



Filter Challenge Test — measures the ability of the filter to provide particle control after the colloidal silica challenge solution has passed through the filter under test.



Integrity Test — tests filter integrity after the challenge test.



Data Analysis and Reporting — calculates the results and reports the data and test conditions.

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 & 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 Background Test

9.3.1 Background testing is required 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.

9.3.2 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.