Integrity Testing Methods

For Membrane Filters

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Integrity Testing

- Principles
- Bubble Point
- Pressure Decay - Pressure Hold / Diffusion
- Automated
Microfiltration Set-up

**Bevigard M**
SOE double O-ring cartridge

**Vitipore II**
SOE, double O-ring cartridge

Inlet → Sample → Drain → Prefilter → Vent

Outlet

Drain → Sample → Final Filter → Vent

Sample
Integrity Test Principles

**Integral Membrane/Device**
- Contaminants larger than rated pore size upstream
- No downstream contamination

**Non-Integral Membrane/Device**
- Contaminants larger than rated pore size upstream
- Downstream contamination
Integrity Test Principles

➔ Which Cartridges?
- Only the membrane (final) filters.
  - Not the prefilters.
Why Perform an Integrity Test?
- Assurance of filter retention and bottled product quality

Method to Detect System Leaks From:
- Improper filter installation
- Damaged filter O-ring or other gasket seal
- Damaged filters from
  - Excessive pressure drop
  - Valve opening/closing speed (pressure hammer)
  - DP during hot CIP on plugged filters
- Chemical attack
When to Perform an Integrity Test?

- Membrane Filters are Installed (New or after long term storage)
  - To ensure proper installation and membrane integrity

- After Every Chemical / Sanitation Procedure
  - Especially if ran at high temperature like 190 F hot water sanitization

- After Bottling Run
  - To ensure integrity throughout the bottling run
Non Destructive - Integrity Test Principles

- The integrity test measures or observes the passage of gas under controlled conditions through a wetted membrane.

- Three types
  - Bubble Point
  - Diffusion
  - Pressure Hold

PVDF microporous Membrane
Integrity Testing

- **Bubble Point**
  - Pressure at which liquid wetting the membrane is extruded out from the pore
Bubble Point is a Function of Pore Size

- For Vitipore II Membrane Cartridges

<table>
<thead>
<tr>
<th>Pore Rating</th>
<th>Bubble Point</th>
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<tbody>
<tr>
<td>1 µm</td>
<td>≥ 9.1 psi</td>
</tr>
<tr>
<td>0.65 µm</td>
<td>≥ 14.2 psi</td>
</tr>
<tr>
<td>0.45 µm</td>
<td>≥ 28.4 psi</td>
</tr>
<tr>
<td>0.22 µm</td>
<td>≥ 45.6 psi</td>
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Bubble Point: Step 1 – WET THE MEMBRANE

- Shut off line pressure after filter wetting/flushing
- Close:
  - Upstream isolation valve
  - Downstream isolation valve (if possible)
- Open:
  - Both housing drains
  - Dome vent
- Water will drain from upstream and downstream housing sides
Bubble Point: Step 2 - Attach Air Supply to Housing

- Close:
  - Upstream housing drain valve
  - Air supply valve
- Attach air supply to either:
  - Dome vent
    - Undo vent assembly first
  - Dome pressure gauge port
  - Upstream pressure gauge port
- Set air pressure regulator to 10 psi
Bubble Point: Step 3 - Attach Hose to Drain Valve - Bucket

- Attach a hose to downstream drain valve
- Submerge the hose open end into a water bucket
Bubble Point: Step 4 - Pressurize Housing

- Open air supply valve
  - Gradually increase pressure regulator, in 5 psi increments
  - Allow pressure to stabilize at each increment
  - Some water will discharge from downstream drain - it is normal
  - In slow increments bring the pressure up to bubble point - 28 psi for a Millipore 0.45µm membrane filter
Bubble Point: Step 4 – Bubble Point

- Small bubbles may appear prior to the bubble point value… this is normal and is due to diffusion.
- Vigorous bubbling should appear at a value > BP … indicating an integral filter set up.
- Vigorous bubbling prior to the bubble point indicates a potential failure.
- If failure occurs, set up should be checked and test run again.

Gradually up to 28 psi

Open

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Integrity Test Principles - Diffusion

- Fully wetted membrane filters retain water in their pores

- When a constant air pressure is applied to the filter:
  - Air will diffuse through the wet filter
  - Upstream air pressure will drop

- Measure air pressure drop on the housing upstream side
Diffusion is a Function of Pore Size

- For Vitipore II Membrane Cartridges

<table>
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<tr>
<th>Pore Rating</th>
<th>Air Diffusion</th>
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<tbody>
<tr>
<td><em>1 μm</em></td>
<td>≤ 12 ml/min at 7.25 psig</td>
</tr>
<tr>
<td><em>0.65 μm</em></td>
<td>≤ 24 ml/min at 9 psig</td>
</tr>
<tr>
<td><em>0.45 μm</em></td>
<td>≤ 45 ml/min at 22 psig</td>
</tr>
<tr>
<td><em>0.22 μm</em></td>
<td>≤ 13.3 ml/min at 41 psig*</td>
</tr>
</tbody>
</table>

- *10 inch cartridge
Integrity Testing

• **Diffusion Flow**
  - Diffusional flow integrity test measures the gas diffusion rate
  - This is typically done at 80% of the bubble point pressure
  - An integral membrane will diffuse at a specific rate (mls/min/cartridge)
Integrity Testing

• Pressure Hold
  – Housing is pressurized to 80% of bubble point
  – Gas supply is turned off
  – Integrity is determined by how fast the pressure drops
Integrity Testing

• Pressure Hold

  – If the pressure drops too fast (i.e. 2 psi in five minutes), than you failed this gross integrity test & troubleshooting should occur.

  – This method is not as accurate as the diffusion test without precise pressure gauges.
Troubleshooting of Upstream Leak

- Loss of pressure due to hardware leak
  - Housing
  - Upstream piping

- Examine the following areas:
  - Dome-to-base housing gasket condition
    - No flat areas, no nicks or abrasion, no “home-made” gaskets
  - Threaded connections
    - Re-seal with Teflon tape
  - Valves
    - Replace valve seat or valve itself
  - Avoid Teflon gaskets
    - EPDM or silicone give better seal
Troubleshooting-Upstream-to-Downstream Leak

- Filters not fully wetted
  - Almost always the cause of this type of problem
  - Re-wet using procedure previously described
- Filters O-rings were rolled or damaged during installation
  - Remove dome and filters to inspect O-rings
- Housing internal O-rings are missing or damaged
  - Remove base plate where filters are installed and inspect
- Filters are not integral
  - Contact Gusmer
  - Save the filters (bag and store in cool place) for return/analysis
Shut Down / Short Term Storage

• Shut Down
  – Push wine from housing with Nitrogen
  – Flush with ambient temperature water in forward direction (integrity test?)
  – Warm water regeneration cycle followed by a sanitation cycle
  – Pressurize housing with a little filtered nitrogen
  – Close valves

System is good for overnight or a weekend
Long Term Storage

• Long Term Storage
  – Most frequently cartridges are removed from the housings
  – O-rings are removed
  – Cartridges are stored in solution of S02 (300-500 ppm) and citric acid (pH 3.5)
**IT Failure & Troubleshooting**

Pressure hold integrity test failure (>5 psi pressure loss after 10 min)

**Tube Test**
Attach narrow bore tubing (~1/4” ID) to downstream side of filters, submerge end in a bucket of water, observe bubbling. If bubbles are:

- **Slow and Bland**
  - Leak is HARDWARE upstream of filters
    - CHECK:
      - Housing Dome O-ring
      - All bleed valves upstream
      - All fitting joints upstream
      - All valve seats upstream
      - All threaded connections (use Teflon tape if leaking)
      * Spraying soapy water on any of these areas will help expose the problem
    - Once upstream leak is found and rectified, repeat Integrity test

- **Vigorous**
  - Leak is FILTER related
    - RE-WET AND RE-TEST
      - Be sure to vent housing and throttle downstream valve to create back pressure of 15-20 psi (especially for newly installed filters)
    - If a failure is still observed, remove the housing dome, CHECK:
      - Filter installation (check to see if any O-rings have rolled up and are visible)
      - Remove filters and check condition of O-rings (one small nick in an O-ring can cause a significant leak)

1. If an O-ring problem is exposed and rectified, re-wet and re-test
2. If there are no O-ring problems observed or the filters still fail the re-wet and re-test in (1), contact your local Millipore Rep, SAVE the filters and return them to Millipore for failure analysis.

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