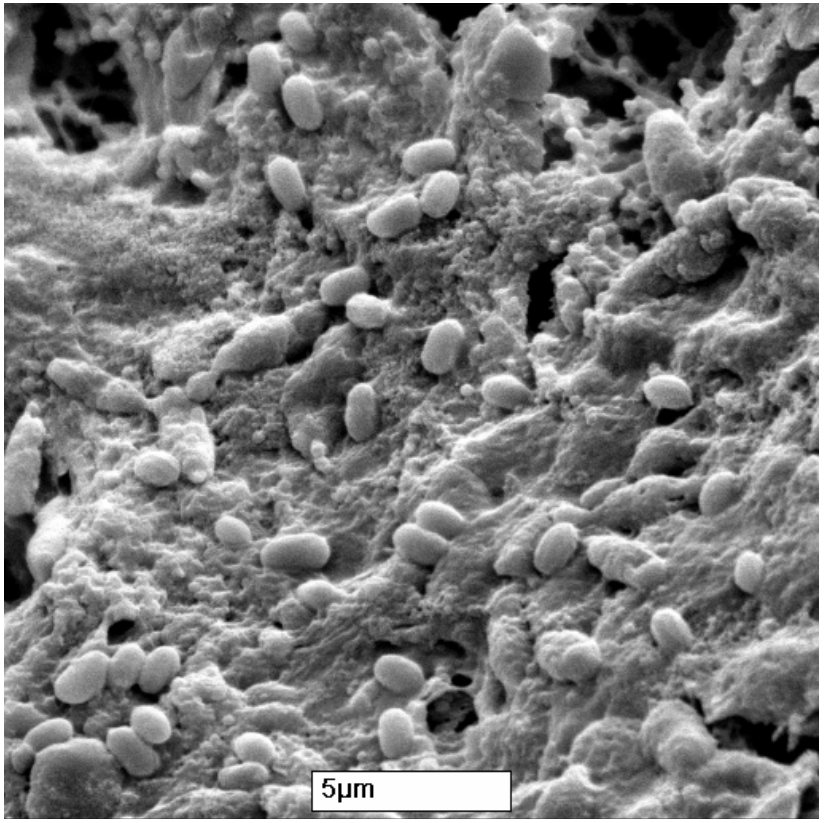
Integrity Testing Methods

For Membrane Filters



2018

Bill Merz - Gusmer

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Integrity Testing

- Principles
- Bubble Point
- Pressure Decay - Pressure Hold / Diffusion
- Automated

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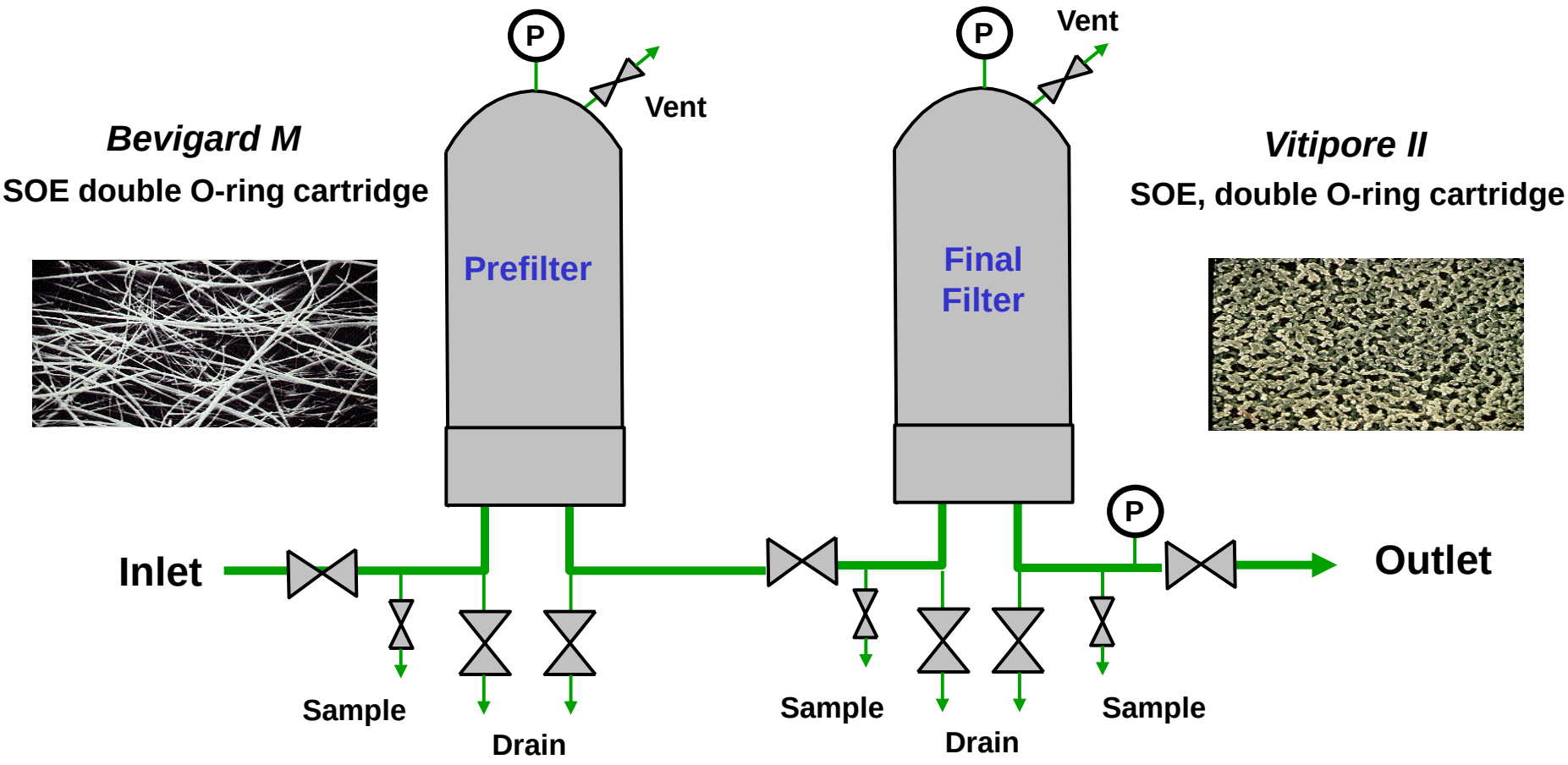
Microfiltration Set-up



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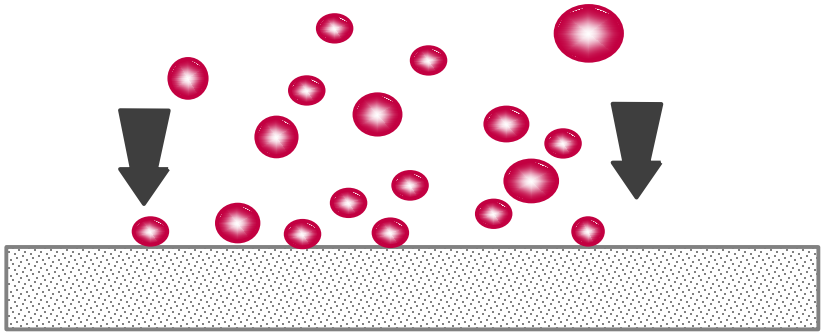
Integrity Test Principles

Integral Membrane/Device

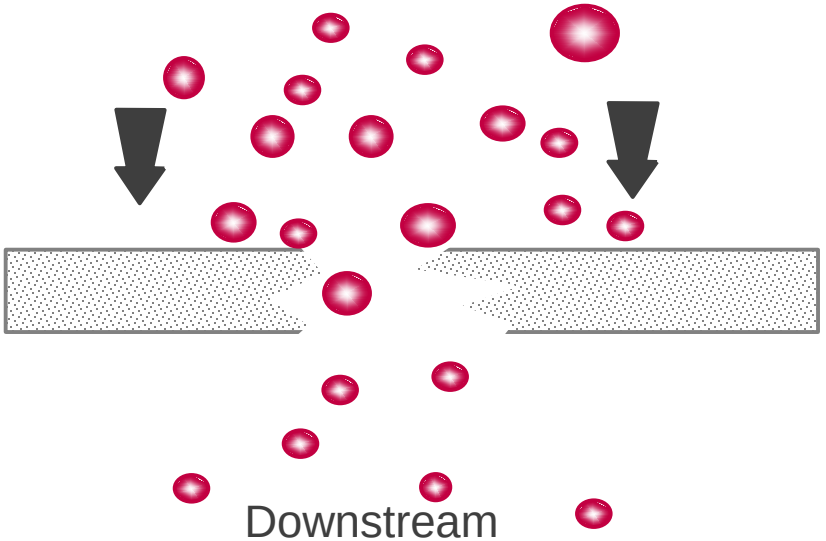
Contaminants larger than rated pore size upstream



No downstream contamination

Non-Integral Membrane/Device

Contaminants larger than rated pore size upstream



Downstream contamination

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Integrity Test Principles

→**Which Cartridges?**

- Only the membrane (final) filters.
  - Not the prefilters.

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Integrity Test Principles

→ **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

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Integrity Test Principles

→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

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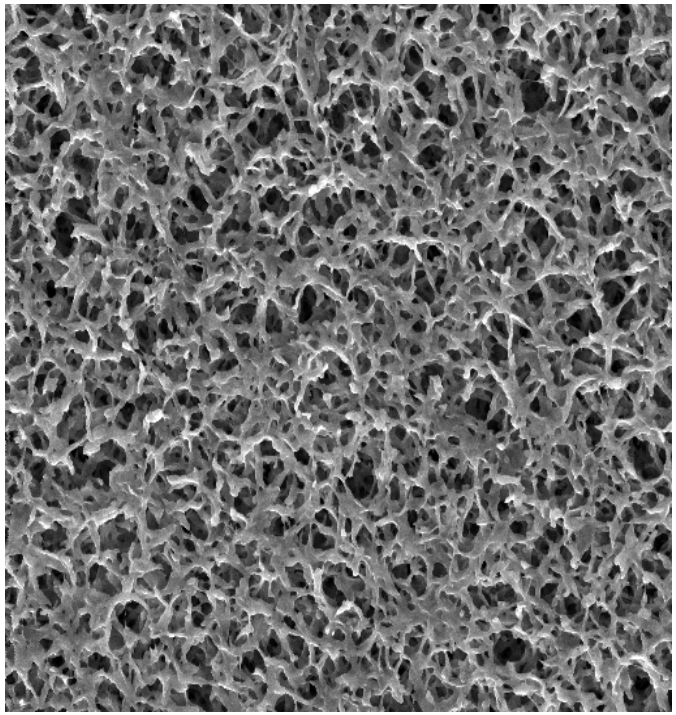
Non Destructive - Integrity Test Principles



PVDF microporous Membrane

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

- Three types
  - Bubble Point
  - Diffusion
  - Pressure Hold

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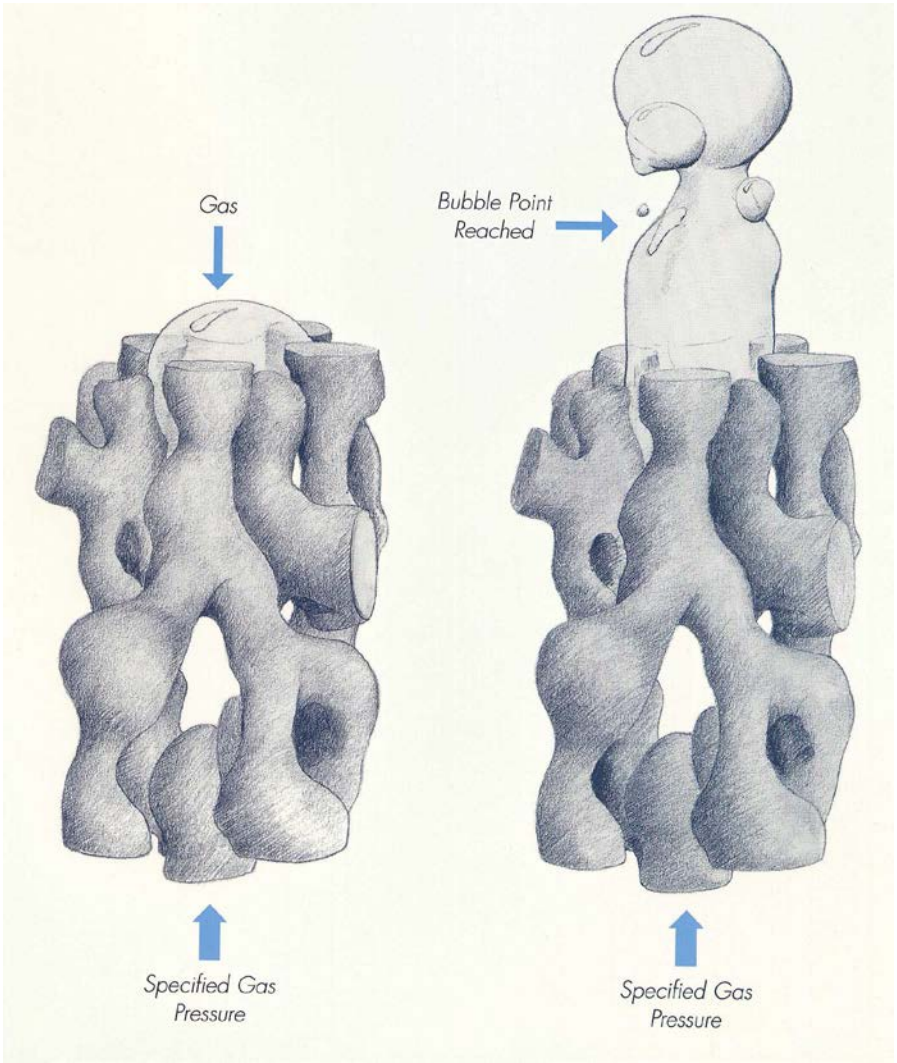
Integrity Testing

- Bubble Point
  - Pressure at which liquid wetting the membrane is extruded out from the pore



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Bubble Point is a Function of Pore Size

- For Vitipore II Membrane Cartridges

Pore Rating	Bubble Point
1 µm	≥ 9.1 psi
0.65 µm	≥ 14.2 psi
0.45 µm	≥ 28.4 psi
0.22 µm	≥ 45.6 psi

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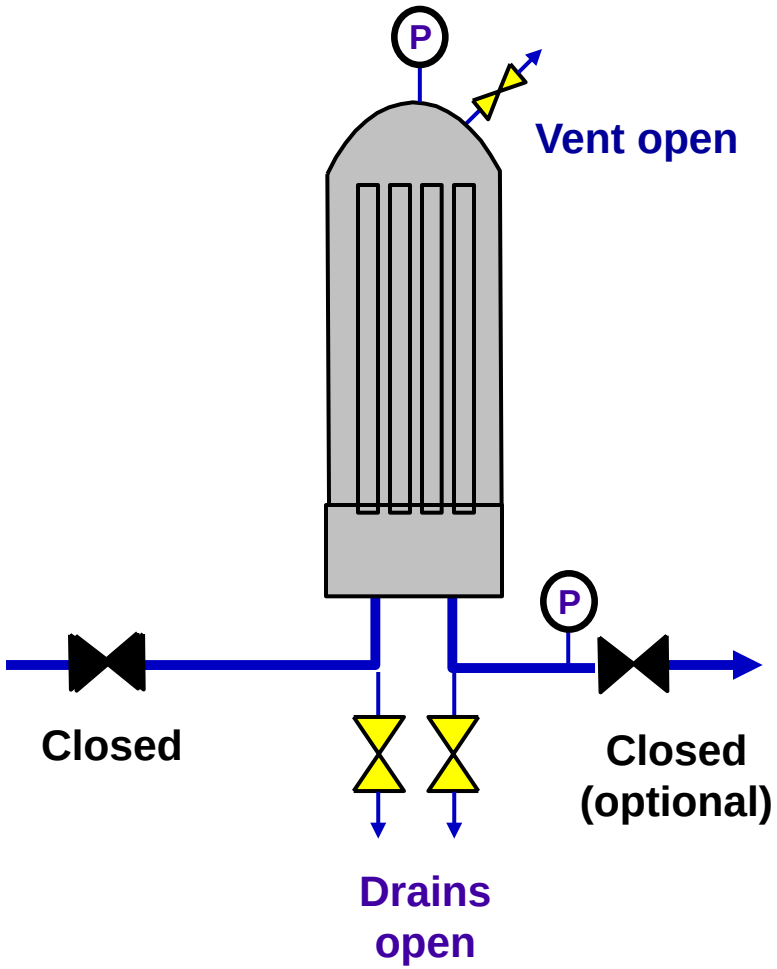
Bubble Point: Step 1 – WET THE MEMBRANE

- 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



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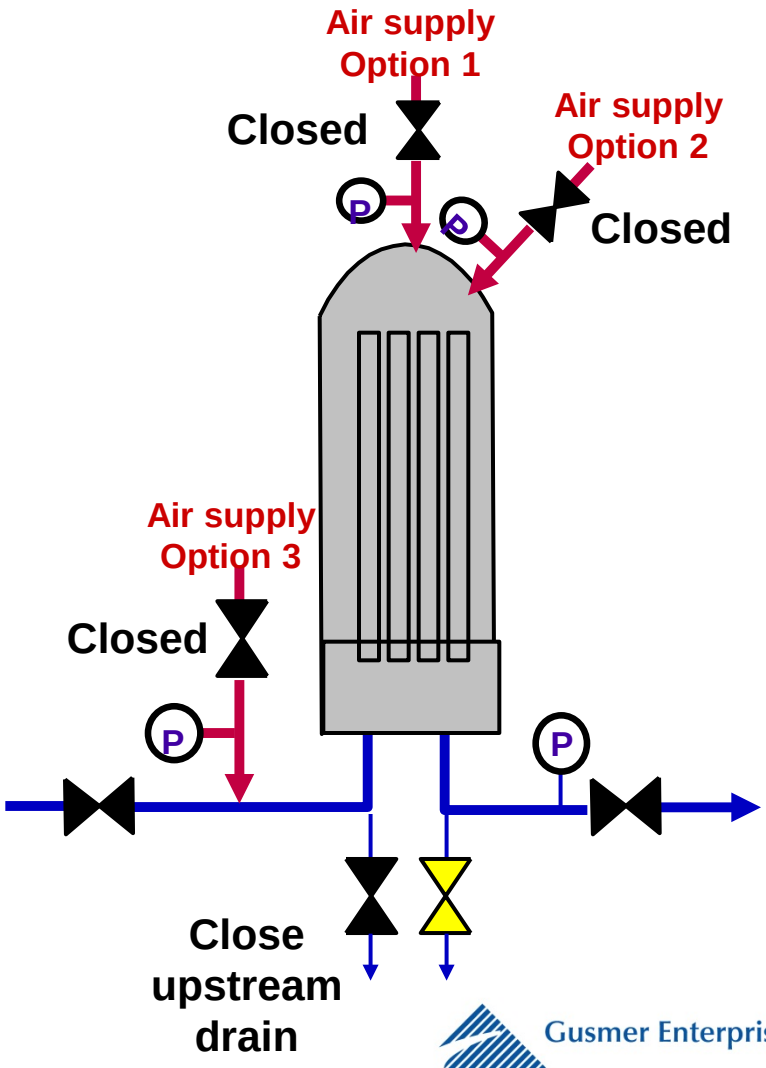
Bubble Point: Step 2 - Attach Air Supply to Housing

- 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



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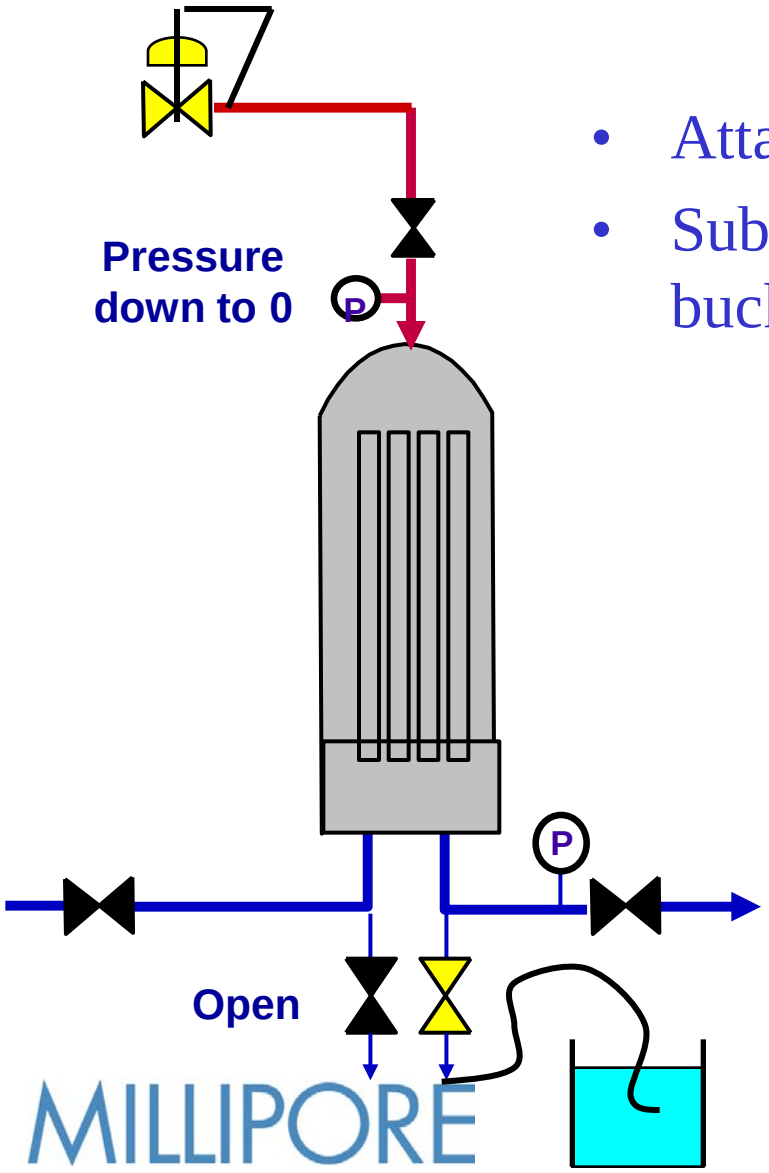
Bubble Point: Step 3 - Attach Hose to Drain Valve - Bucket



- Attach a hose to downstream drain valve
- Submerge the hose open end into a water bucket

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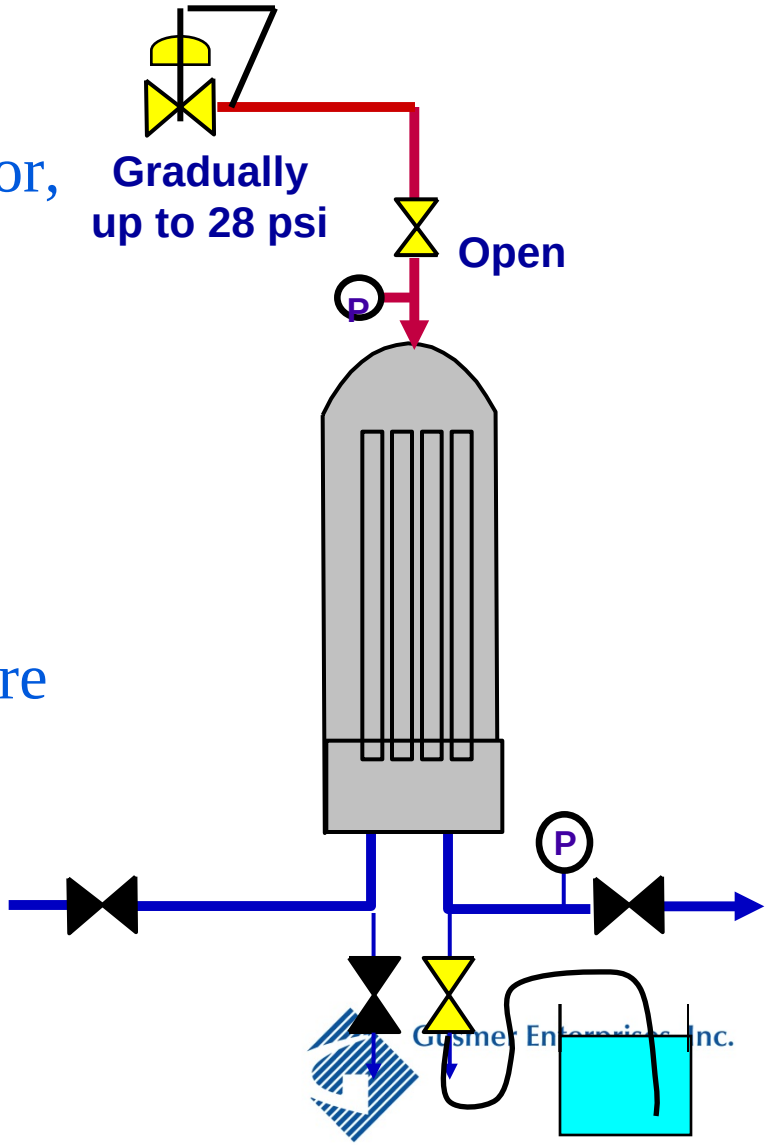
Bubble Point: Step 4 - Pressurize Housing

- 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



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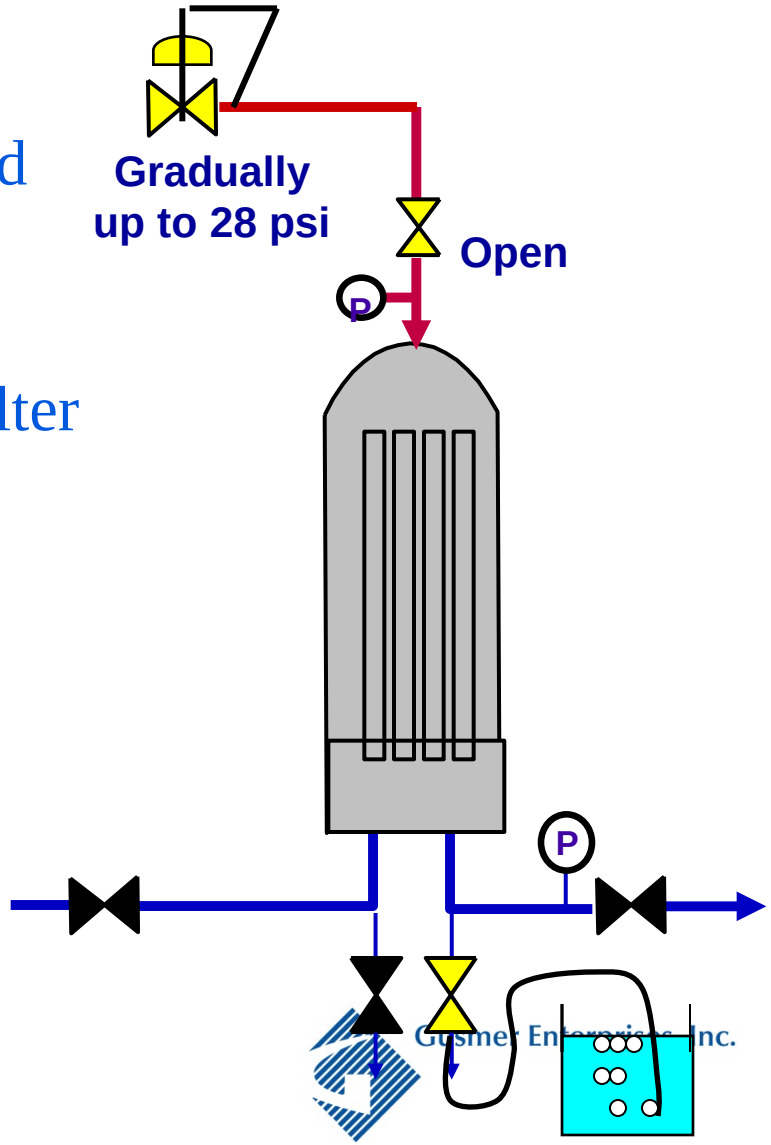
Bubble Point: Step 4 – Bubble Point

- 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.



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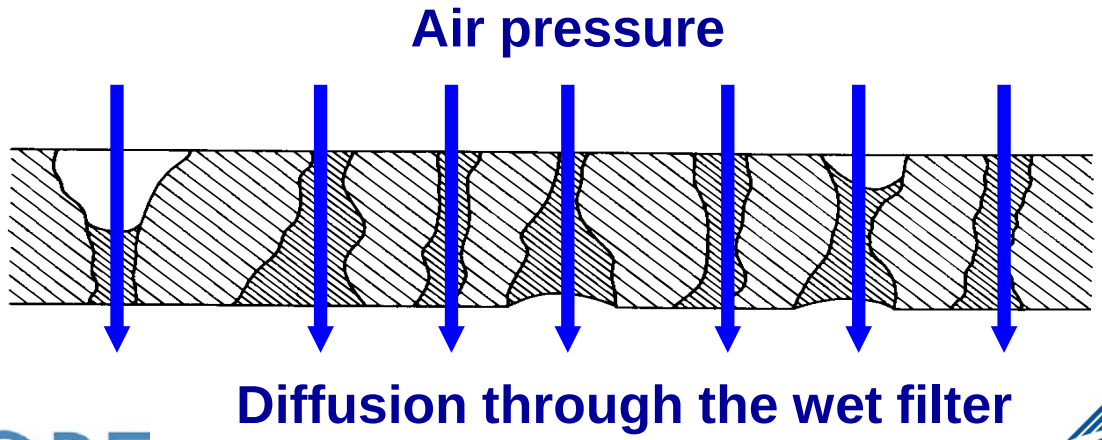
Integrity Test Principles - Diffusion

→ 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



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Diffusion is a Function of Pore Size

- For Vitipore II Membrane Cartridges

Pore Rating	Air Diffusion (30 inch at 23-25 c)
1 µm	≤ 12 ml/min at 7.25 psig
0.65 µm	≤ 24 ml/min at 9 psig
0.45 µm	≤ 45 ml/min at 22 psig
0.22 µm	≤ 13.3 ml/min at 41 psig*

- * 10 inch cartridge

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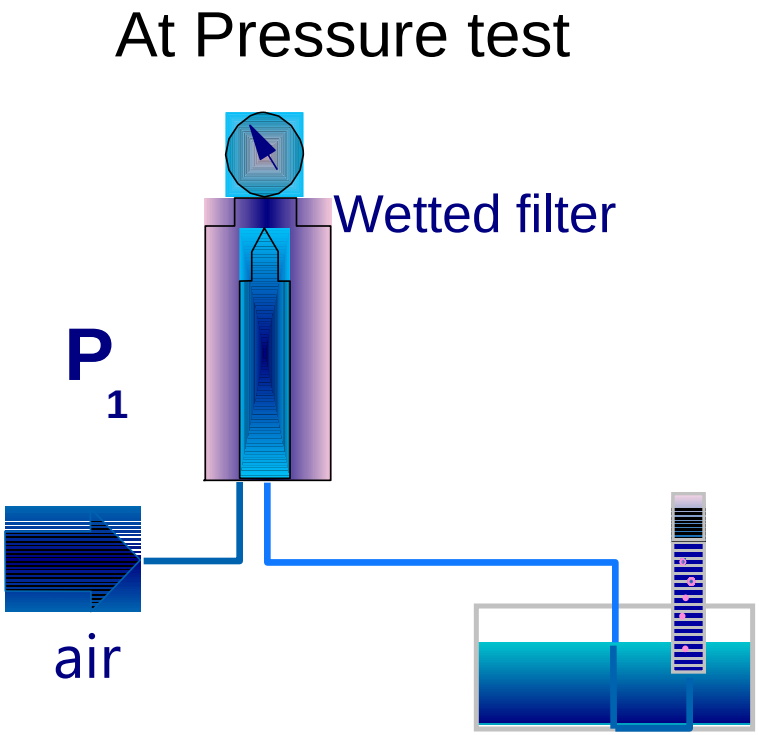
Integrity Testing

- 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)



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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

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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

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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

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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

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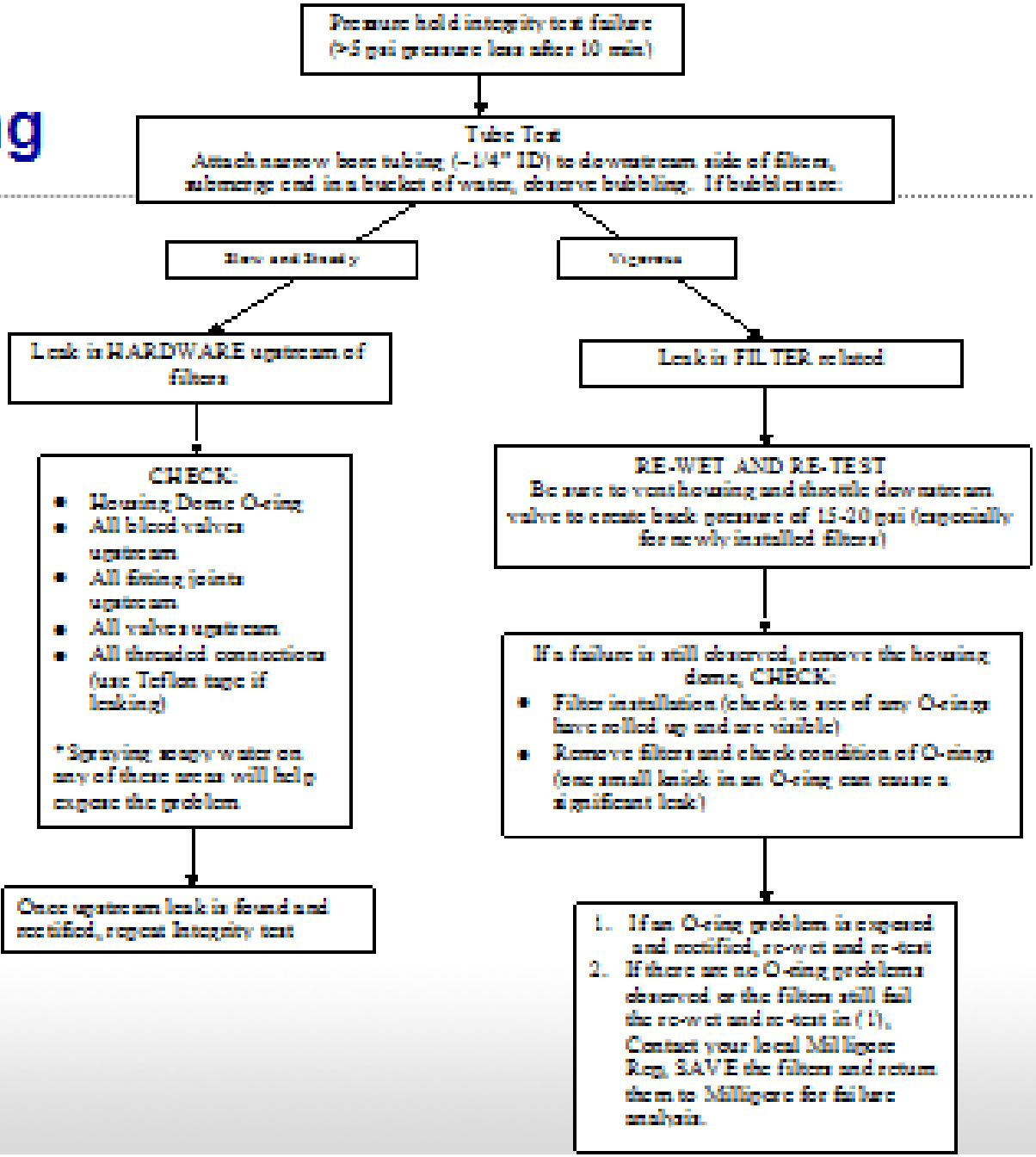
Long Term Storage

- 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



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