Pall Corporation

Contact Us: www.pall.com/contact

Eliminating the Need to Prewet Hydrophobic Filters with Low Surface Tension Fluids

Eliminating the Need to Prewet Hydrophobic Filters with Low Surface Tension Fluids

J. O'Sullivan, Ph.D.
B. Gotlinsky, Ph.D.

Filter cartridges constructed from all fluorocarbon components are employed to filter high purity chemicals in today's semiconductor fabrication processes. The strict requirements for clean materials, as well as compatibility issues, often necessitate the use of fluorocarbon materials. PTFE is an excellent material for filter membranes and is widely used for bulk, distribution, and point-of-use applications.

Many applications involve the filtration of aqueous chemicals, which include etching, cleaning, photoresist development, etc. Since the PTFE membranes are hydrophobic, prewetting with low surface tension fluids such as isopropanol (IPA) is required. Prewetting can be best accomplished by vacuum-drawing or pumping the low surface tension fluid through the filter. This is especially recommended for 0.05 µm filters, which are more difficult to wet due to the tighter pores. While the prewetting procedure is well established, recent environmental regulations limiting the amount of organic vapors in the workplace air are forcing users to reduce the utilization of IPA and other solvents for prewetting purposes. The permissible exposure limit (PEL) established by OSHA and the threshold limit value (TLV) established by ACGIH for IPA is 400 ppm.

Attempts at prewetting the hydrophobic filters by submerging the filters in water pressurized above the bubble point of the membrane has had limited success. Until now, providing hydrophobic filters prewet so that they can be installed directly into tools and distribution systems has been hampered by packaging and fluid shipment and purity issues.

Test Methods and Results

Validation of Manufacturing Process
In order to validate the manufacturing process, Kilit* ampules were used to determine the extent of the heat treatment required to provide sterile filter environment. The Kilit ampules, manufactured by Becton Dickinson, are biological indicators used in-situ of a sterilizing cycle. The glass ampules contain suspensions of *Bacillus stearothermophilus* ATCC* 7953 in a culture medium. After the sterilization process, if any viable microorganisms survive, they produce acid during growth upon incubation. The acid produced will change the color of the bromcresol purple indicator contained in the ampule from purple to yellow, providing an extremely sensitive measure of sterilization efficacy. It should be noted that this is a very aggressive test, as the biological concentration of the Kilit ampule far exceeds that found in UHP water. Therefore, if no viable organisms remain in the Kilit after processing, this allows a high safety factor for sterilization of the UHP water.

During manufacturing trials, Kilit ampules were placed in the cores of filters processed. After the sterilization process, the Kilit ampules were incubated at 60°C for 7 days, with observations for turbidity and color change, which would signify growth, at 24 hours, 48 hours, and at 7 days. For the final process used to manufacture the filters, no viable microorganisms were found in the Kilit ampules, validating the process to yield a sterile packaging environment for the filter.

Top

Sterility of Water in Packaged Filters

In order to assess shelf life, packaged prewet filters were kept in storage for 3, 6, 9, 12, 18 and 24 months, and the water sampled for viable bacteria using Mueller Hinton Agar and R2A Agar as growth media. The water samples were incubated in the growth media for 5 days at 25°C, and examined for growth of colonies.

Sampling of the ultrapure water contained in the packaging at intervals of 3, 6, 9, 12, 18 and 24 months shelf life for microbial growth resulted in no viable organisms detected. Table I summarizes the data.

Table I

Viable Organisms After 5 Day Incubation at 25°C		
Shelf Life Period	**Mueller Hinton Agar**	**R2A Agar**
3 months	0	0
6 months	0	0
9 months	0	0
12 months	0	0
18 months	0	0
24 months	0	0

This confirms the guaranteed shelf life of 12 months with respect to sterility. The sterility of the samples during shelf life means that microbial contamination will not occur, eliminating the need for a preflush or sanitization of the filters prior to being used on line.

Top

Stability of Prewet Condition

To determine the effect of shelf life on prewet condition, the filters from the shelf life testing noted in the above paragraph were subjected to differential pressure measurement. If the filters were not completely prewet, high differential pressures would be observed. Differential pressure was measured by flowing the UHP water through the filters at varying flow rates, both ascending and descending, and measuring differential pressure using a pressure transducer. Flow was monitored with a rotameter.

By measuring the differential pressure in UHP water directly out of the package, stability of the prewet condition was confirmed through one year shelf life. The differential pressure data also confirmed that the prewet process had no effect as compared to conventionally prewet cartridges. The differential pressure data are summarized in Table II.

Table II

Differential Pressure (psid/gpm)		
Shelf Life Period	**0.01 µm Prewet Emflon® PF filter**	**0.05 µm Prewet UltiKleen™ filter**
3 months	0.85	0.75
9 months	0.85	0.75
12 months	0.85	0.75
24 months	0.85	0.75

The differential pressures measured indicate that dewetting of the hydrophobic membrane did not occur over the 24 month shelf life testing.

Top

Purity of UPW from Bags Containing Prewet Filters

The 18 M -cm UPW employed for filter flush and packaging is semiconductor grade DI water, containing only trace levels of ionic contaminants as determined via ion chromatography. Since the Emflon® PF filter and the UltiKleen™ filter are constructed solely of fluorocarbon materials, as is the packaging materials, minimal contamination, other than fluoride ions, are expected to leach into the water, even with long term exposure.

Sampling of UPW from bags containing prewet Emflon® PF filters yielded the following levels of ionic species in the water, as determined utilizing Dionex ion chromatographs:

Specie	Level (ppb)
F^-	low single digit ppm
Cl^-	single digit
NO_3^-	single digit
HPO_4^{2-}	single digit
SO_4^{2-}	single digit
Na^+	low double digit
K^+	low double digit
Mg^{2+}	single digit
Ca^{2+}	low double digit
Fe^{2+}	single digit
Cu^{2+}	single digit
Ni^{2+}	single digit
Zn^{2+}	single digit
Co^{2+}	single digit

Since the UPW is of high purity, start up with the filter is rapid.

Top

TOC Rinse-up of all Fluorocarbon Filters

A 0.05 µm Emflon® PF filter (AB1F00053EH1) was prewet by vacuum drawing 3 liters of 60:40 IPA:water through the filter. The filter was subsequently completely submerged in 60:40 IPA:water for 30 minutes. At the end of the 30 minute soak the filter was removed from the 60:40 IPA:water and the excess fluid was allowed to drain off. The 0.05 µm Emflon PF filter was then installed in a filter housing and DI water flow was initiated. The DI water flowrate was set at 2 GPM and the TOC levels upstream and downstream of the housing were monitored with Anatel TOC analyzers. The A-1000 TOC analyzer was bypassed until 15 minutes after initiating DI water flow. The latter precaution was taken so as to avoid contaminating the analyzer with high concentrations of alcohol. The TOC readings upstream and downstream of the 0.05 µm Emflon PF filter during the DI water flush are shown in Figure I. The downstream TOC level was observed to be ~ 10 ppb after 90 minutes of commencing DI water flow. The downstream TOC was within 3 ppb of the upstream TOC after 3 hours of DI water flow at 2 GPM.

In the case of the 0.05 µm Prewet Emflon PF filter (AB1F00053EH1K3) the filter was removed from its packaging and placed in the test filter housing. Upon installation of the filter the DI water flow was initiated (2GPM) and the TOC levels were monitored continuously upstream and downstream of the filter. The downstream TOC level was less than 10 ppb within ten minutes and within 2 ppb of the upstream TOC level within 15 minutes- see Figure I. The downstream TOC level was within 0.5 ppb of the upstream level within 25 minutes.

The results outlined above indicate that prolonged flushing times are required to remove the residual alcohol remaining after the prewetting process. In the rinse-up testing outlined above, an additional 330 gallons of 18 MΩ-cm DI water was required for the Emflon PF filter prewet by conventional methods to reach 3 ppb above background levels compared to the Prewet Emflon PF filter. The Prewet Emflon PF filter not only eliminates the need for employing low surface tension fluids to prewet the medium but drastically reduces the time required to change out all-fluorocarbon filters. In addition the Prewet Emflon PF filter and the Prewet UltiKleen filter eliminate the possibility of cross contamination by the prewetting fluid.

Top

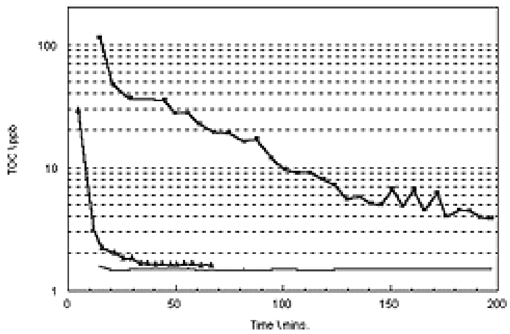
Conclusions

The availability of hydrophobic filters which are supplied prewet has definite advantages, including
• Eliminating the need to prewet with low surface tension fluids such as isopropanol (IPA). The elimination of the prewet process will reduce operator exposure to alcohols, negate the need for grounding systems due to flammability hazards, and reduce waste disposal costs.
• Reduce the time required to change out all-fluorocarbon filters due to significantly faster TOC rinse-up times with the ability to immediately install the filters in bulk, distribution, and POU applications. The

rapid rinse-up times will result in significant savings in volume of UPW required to achieved background TOC levels.
• Eliminate the possibility of cross contamination due to inadequate flushing with DI water to remove the prewetting solvent.
• Eliminate the possibility of experiencing a high clean differential pressure due to incomplete wetting of the hydrophobic filter.

Figure I

Downstream TOC Levels During Rinse-up for a Emflon® PF filter (•) immediately after prewetting with 60/40 IPA/water and a Prewet Emflon® PF filter (Δ). The upstream TOC levels (—) are as shown.



Top

References

* Kilit is a trademark of Becton Dickinson and Company.
ATCC is a trademark of the American Type Culture Collection.

Top