The Study of Effectiveness of Key Priming Cycles in IntelliGen® ULV

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As semiconductor devices have been miniaturized, high requirement of impurity control has become essential. To respond to this demand, a very tight pore size point-of-use filter has been developed. Although tight pore size filters are effective in terms of impurity removal, it comes with the difficulty of filter preparation. A filter that is not properly prepared or in other word not properly primed will release residual bubbles overtime once photoresists pass through. The consequence of this is to scrap wafers due to defects caused by bubbles.

Two-stage dispense system IntelliGen® ULV developed to have the capability to fill tight pore size membranes. It featuring totally six technologies that enhance the elimination of bubbles in micro and nano size. The six technologies are: 1. soak, 2. backflush to vent, 3. backflush to inlet, 4. filterflush with filtration pressure of 2 psi, 5. filterflush with filtration pressure of 20 psi, and 6. flush bubbles. The experiment data showed that three technologies among six (Backflush to vent, filterflush, and flush bubbles) gave a promising priming performance. The evaluation results of the four recipes passed criteria defined by Entegris.

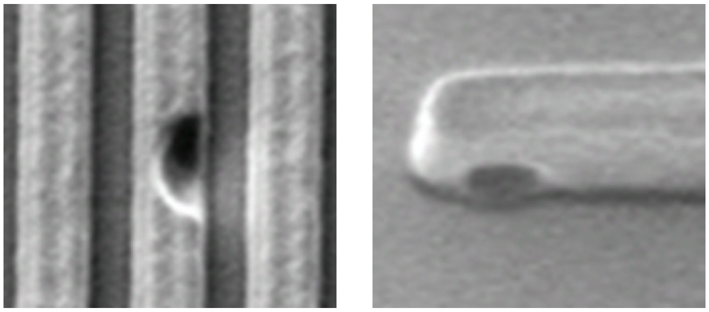


Figure 1. Defect image courtesy of KLA tensor

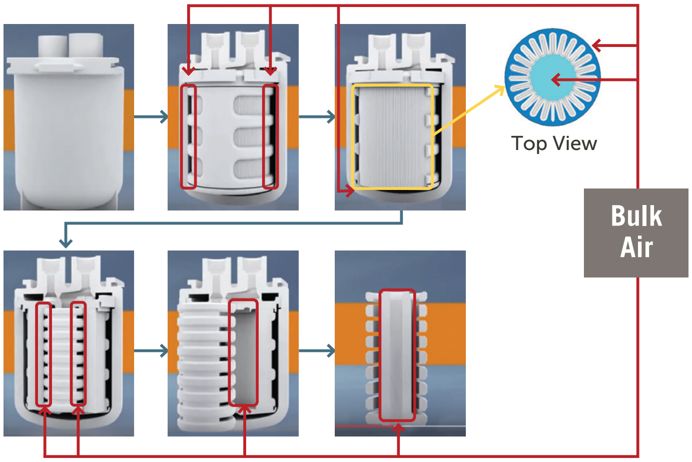


Figure 2. Location where bulk air exists

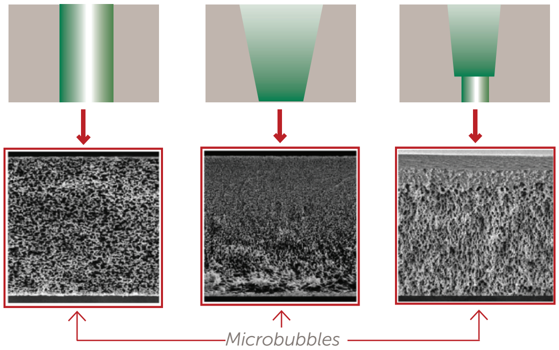


Figure 3. Location where microbubbles exist

SCOPE OF THE EXPERIMENT

The scope of the experiment is to compare the effectiveness of each priming technology. Priming recipe is set with seven steps of priming cycle. The priming cycles are sequenced in the order that bulk air is first removed then followed by microbubbles and nanobubbles at last. Step 1 to Step 2 is to eliminate bulk air, Step 3 to Step 5 is to eliminate microbubbles and Step 6 to Step 7 is to eliminate nanobubbles. The six priming technologies to be evaluated is set at Step 6 in the recipe as a selectable step. Step 6 is varied by changing priming technology. The amount of bubbles after priming completes is then observed by liquid particle counter and is used for the judgement of effectiveness of each priming technology.

RECOMMENDED PRIMING SEQUENCE

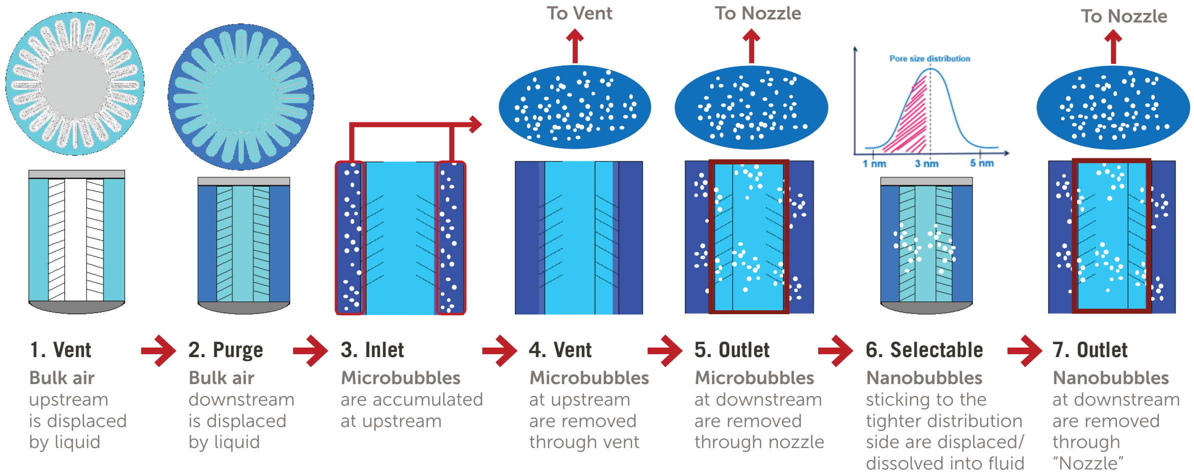


Figure 4. Sequence of seven priming cycles in the evaluation of effectiveness of the six priming technologies

Table 1. Summarization of the seven steps in recommended priming sequence and the location where selectable cycles are added (FR is filtration rate and FP is filtration pressure)

STEP	RECIPE 1	RECIPE 2	RECIPE 3	RECIPE 4	RECIPE 5	RECIPE 6	RECIPE 7	TARGET
1	Vent	Vent	Vent	Vent	Vent	Vent	Vent	**Bulk air**
2	Purge	Purge	Purge	Purge	Purge	Purge	Purge	**Bulk air**
3	Inlet FR = 0.5 mL/s, FP = 20 psi	Inlet FR = 0.5 mL/s, FP = 20 psi	Inlet FR = 0.5 mL/s, FP = 20 psi	Inlet FR = 0.5 mL/s, FP = 2 psi	Inlet FR = 0.5 mL/s, FP = 20 psi	Inlet FR = 0.5 mL/s, FP = 20 psi	Inlet FR = 0.5 mL/s, FP = 20 psi	**Microbubble**
4	Vent	Vent	Vent	Vent	Vent	Vent	Vent	**Microbubble**
5	Outlet FP = 20 psi	Outlet FP = 20 psi	Outlet FP = 20 psi	Outlet FP = 20 psi	Outlet FP = 20 psi	Outlet FP = 20 psi	Outlet FP = 20 psi	**Microbubble**
6	Soak	Backflush to Vent	Backflush to Inlet	Filter flush FP = 2 psi	Filter flush FP = 20 psi	Flush bubbles	Filter flush FP = 20 psi 10 cycles	**Nanobubble**
7	Outlet FP = 2 psi	Outlet FP = 2 psi	Outlet FP = 2 psi	Outlet FP = 2 psi	Outlet FP = 2 psi	Outlet FP = 2 psi	Outlet FP = 2 psi	**Nanobubble**

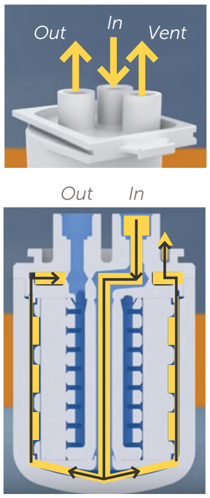
Filter - Impact 8G UPE UC 3.0 nm, Liquid - OK73

- Fixed portion of recipe
- Selectable portion of recipe

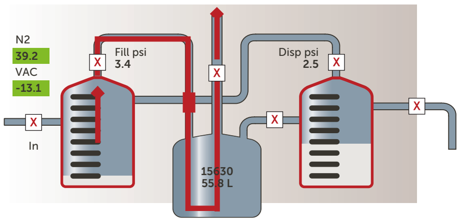
OPERATING SEQUENCE OF EACH PRIMING CYCLE

Standard Cycles

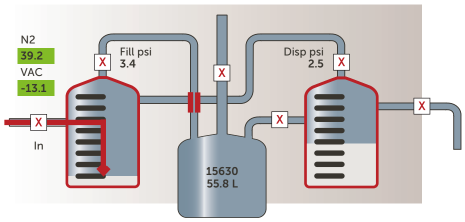
Vent cycle. The vent cycle is for the elimination of bulk air at filter upstream. There are two segments in vent cycle: venting and filling. Liquid goes into filter via inlet port and leave filter via vent port. Vent cycle is recommended to use at the first step to eliminate bulk air at filter upstream.



Impact® 8G Filter



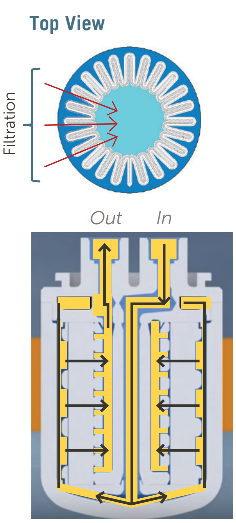
1. Venting
Bulk air upstream is removed.



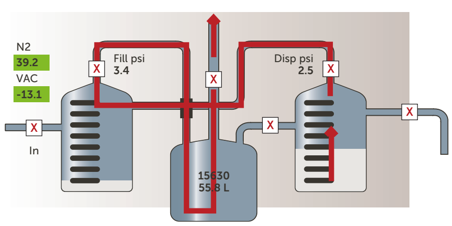
2. Filling
Recharge liquid for the next cycle.

Figure 5. Vent cycle

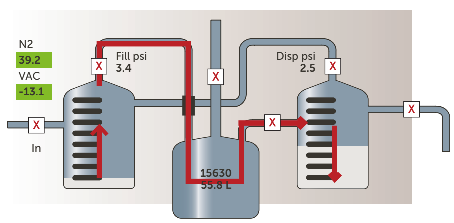
Purge cycle. The purge cycle is for the elimination of bulk air downstream. There are three segments in purge cycle: purging to vent, filtration, and purging to inlet. Purge cycle is recommended to use at the second step to eliminate bulk air at filter downstream.



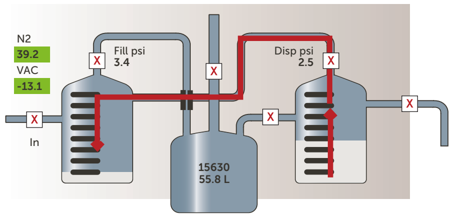
Impact 8G Filter



1. Purging to Vent
Bulk air in purge line is removed through vent.



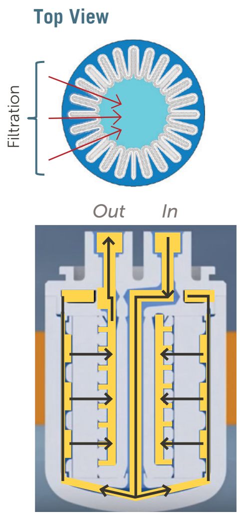
2. Filtration
Microbubble in membrane's pores are pushed to dispense chamber.



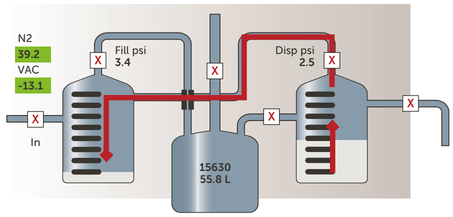
3. Purging to inlet
Microbubbles in dispense chamber are pushed back to fill chamber and are eliminated through vent afterwards.

Figure 6. Purge cycle

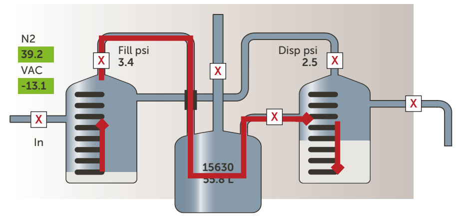
Inlet cycle. The inlet cycle is for the elimination of microbubble in membrane's pores without wasting chemical. There are two segments in inlet cycle: purging to inlet, and filtration. Inlet cycle is recommended to use at the third step to eliminate microbubbles in membrane pores. Those microbubbles will accumulate in fill chamber so vent cycle is recommended to use after inlet cycle to eliminate accumulated microbubbles through vent.



Impact 8G Filter



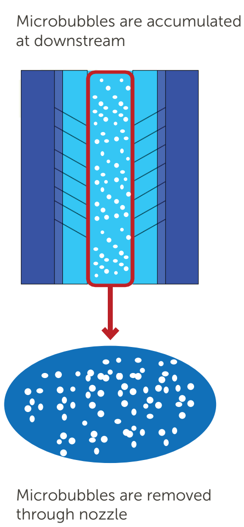
1. Purging to inlet
Microbubbles in dispense chamber are pushed back to fill chamber.

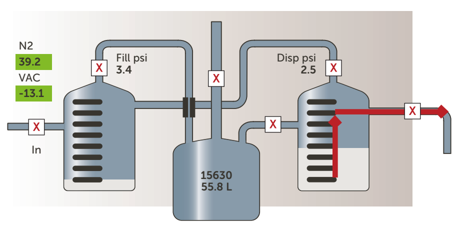


2. Filtration
Microbubbles in membrane's pores are pushed to dispense chamber.

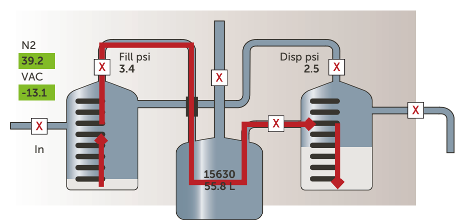
Figure 7. Inlet cycle

Outlet cycle. There are three segments in outlet cycle: 10 mL dispense, normal filtration, and filling. Outlet cycle is recommended to use at the end of priming sequence to completely eliminate microbubble through nozzle.

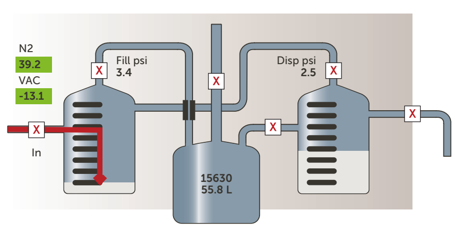




1. 10 mL dispense
Remove bubbles in dispense chamber to nozzle.



2. Normal filtration
Pump starts a recharge for the next cycle.



3. Filling
Liquid is pulled into pump for the next dispense.

Figure 8. Outlet cycle

OPERATING SEQUENCE OF EACH PRIMING CYCLE

Selectable Cycles

Selectable cycle. A selectable cycle is the priming cycle featuring technologies that enhance the elimination of microbubbles and nanobubbles. It is recommended to use after the elimination of bubbles is completed to a certain level. An addition of selectable cycle after outlet cycle in the recommended sequence of priming cycles enables a liquid to displace residual air in micropores and nanopores. There are five selectable cycles: soak cycle, backflush to vent cycle, backflush to inlet cycle, filterflush cycle, and flush bubbles cycle.

Pore size distribution. Membrane pores are usually not uniformly dispersed. There are pores with smaller size and there are pores with larger size than the average. The smaller pores remain dry while bigger pores can be filled by liquid because a liquid chooses to flow through the channel which has less resistance. The selectable cycle can force the liquid by pressure to go through and fill smaller pores.

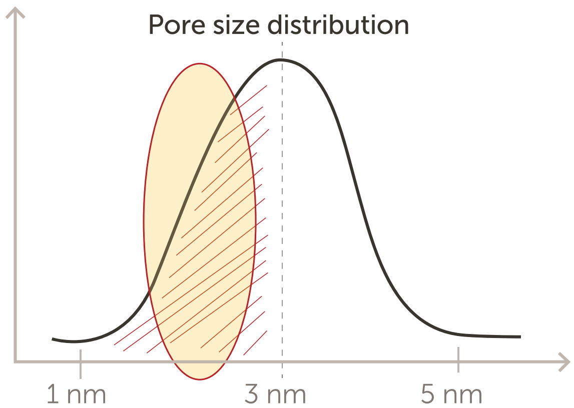


Figure 9. Histogram demonstrates pore size distribution of 3 nm membrane

Soak cycle. There are three segments in soak cycle: venting, soaking, and venting. Liquid is pressurized into membrane pores from upstream. Air escapes from pores can be eliminated during the last venting segment.

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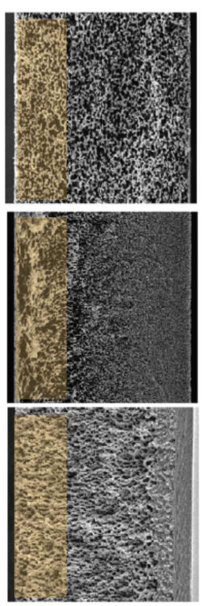
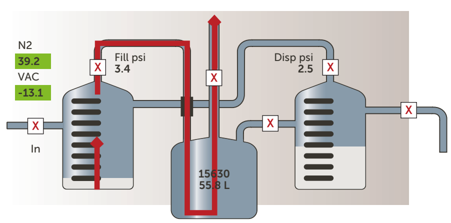
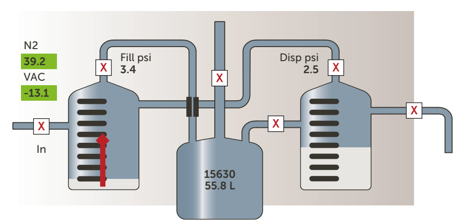


Refers to area where pores can be effectively wetted



1. Venting
Remove upstream microbubbles through vent.

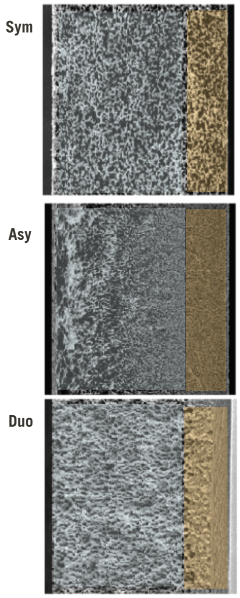


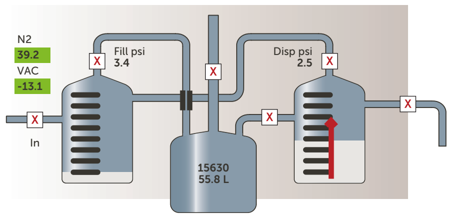
2. Soaking
Pressurize from upstream to enable liquid to penetrate membranes' pores.

3. Venting
Remove microbubbles counterflow backwards to fill chamber.

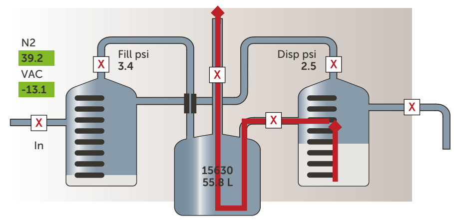
Figure 10. Soak cycle

Backflush to vent cycle. There are three segments in backflush to vent cycle: soaking backwards, filtration backwards to vent, and normal filtration. Liquid is pressurized into membrane pores from downstream. Tight pores at downstream can be filled by liquid and microbubbles inside tight pores can be immediately eliminated through vent during filtration backwards to vent segment.

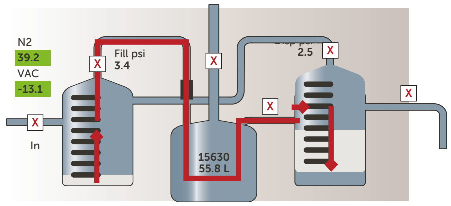




1. Soaking backwards
Pressurize from downstream to enable liquid to penetrate membranes' pores and dissolves nucleation site on membrane surface into liquid.



2. Filtration backwards to vent
Eliminate the liquid with dissolved nucleation site through vent.



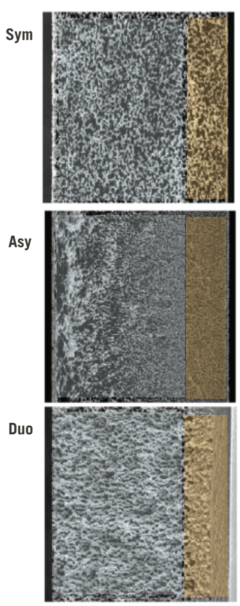
3. Normal filtration
Push air bubbles to dispense chamber.

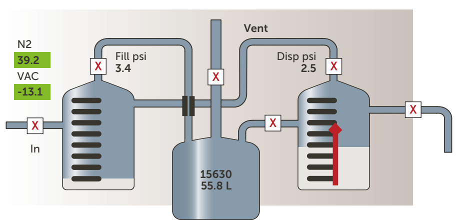
Refers to area where pores can be effectively wetted

Refers to area where pores can be moderately wetted

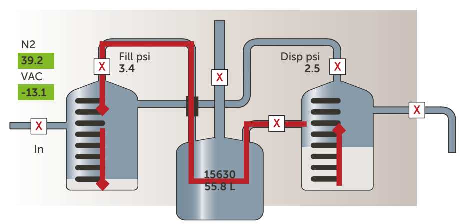
Figure 11. Backflush to vent cycle

Backflush to inlet cycle. There are four segments in backflush to inlet cycle: soaking backwards, filtration backwards to inlet, normal filtration, and purging to inlet. Liquid is pressurized into membrane pores from downstream. Tight pores at downstream can be filled by liquid. Microbubbles inside tight pores are accumulated in fill chamber during purging to inlet cycle.

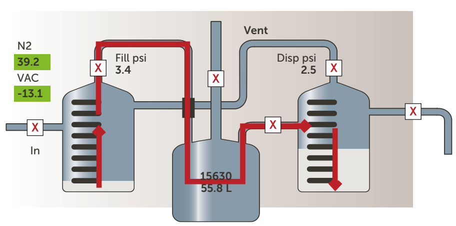




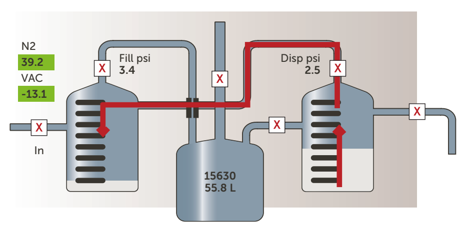
1. Soaking backwards
Pressurize from downstream to enable liquid to penetrate membranes' pores.



2. Filtration backwards to inlet
Liquid is pushed back through membranes' pores to fill chamber.



3. Normal filtration
Liquid is again passed through membrane's pores.



4. Purging to inlet
Microbubbles are collected in fill chamber.

Refers to area where pores can be effectively wetted

Refers to area where pores can be moderately wetted

Figure 12. Backflush to inlet cycle

Filterflush cycle. There are four segments in filterflush cycle: venting, filtration backwards to inlet, normal filtration, and filling. Liquid flow through membrane pores backwards and forwards. Pores throughout membrane can be filled by liquid. Microbubbles inside membrane are accumulated in dispense chamber by normal filtration segment.

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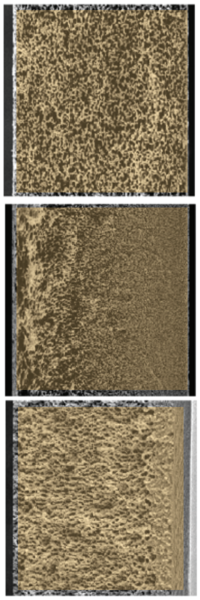
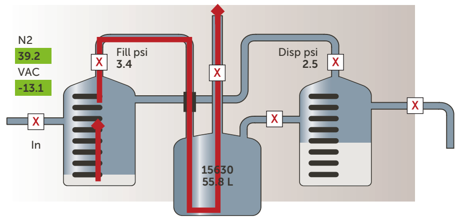
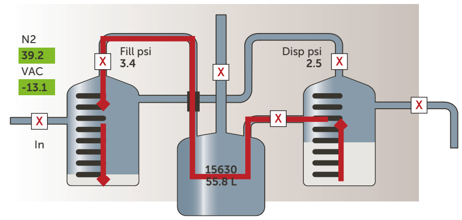
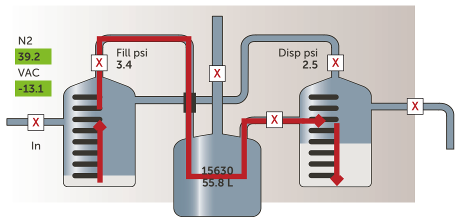




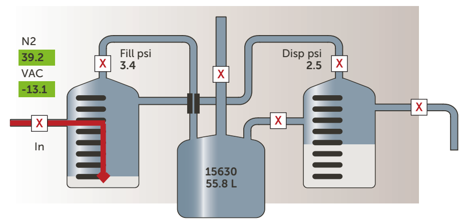
1. Venting
Remove upstream microbubbles through vent.



2. Filtration backwards to inlet
Liquid is pushed back through membranes' pores to fill chamber.



3. Normal filtration
Liquid is again passed through membrane's pores.



4. Filling
Recharge liquid for the next cycle.

Refers to area where pores can be effectively wetted

Figure 13. Filterflush cycle

Flush bubbles cycle. There are four segments in flush bubbles cycle: soaking backwards, filtration to outlet, 10 mL dispense, and normal filtration. Liquid is pressurized into membrane pores from downstream. Tight pores at downstream can be filled by liquid and microbubbles inside tight pores can be immediately eliminated through outlet during 10 mL dispense segment.

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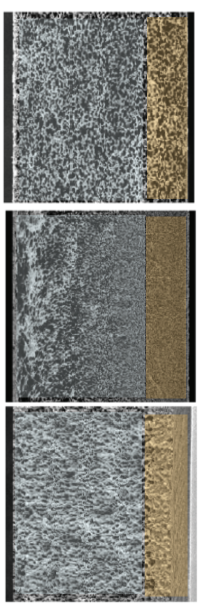
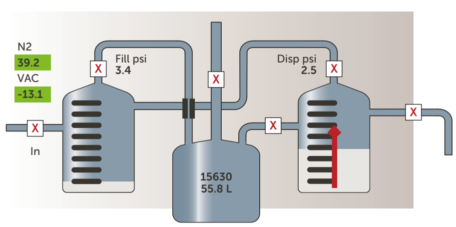
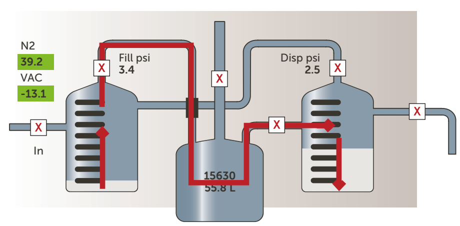
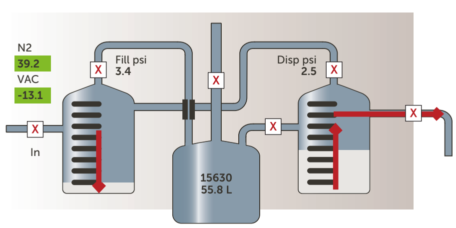




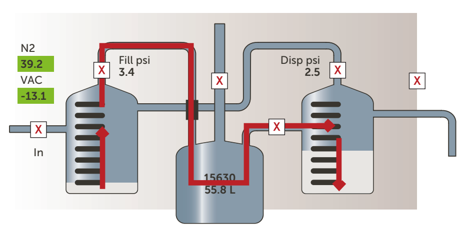
1. Soaking backwards
Pressurize from downstream to enable liquid to penetrate membranes' pores.



2. Filtration to outlet
Outlet valve opens and liquid is pushed through membrane pores and bubbles are brought to dispense chamber.



3. 10 mL dispense
Bubbles come with liquid during filtration are removed to nozzle.



4. Normal filtration
Pump starts a recharge for next dispense by starting normal filtration segment.

Refers to area where pores can be effectively wetted

Refers to area where pores can be moderately wetted

Figure 14. Flush bubbles cycle

METHOD

Experiment setup. An IntelliGen ULV pump with firmware "Released M1004.798" and installed with an Impact 8G UC UPE 3.0 nm filter was used. PGMEA was used as the dispensed fluid. After priming, the dispensed liquid is stored in the 30 mL cylindrical collection vessel before being drawn into the particle counter by the syringe sampler. After passing through the particle counter, the liquid is returned to the bottle. Particle counter models Rion®KS41-A measuring 0.15 microbubbles were used. A sample flow of 1.0 mL at 2.0 mL/s was measured every 30 seconds and recorded over 200 cycles after completion of priming.

Experimentation. A baseline of liquid particle counter is established every time before each evaluation starts. The baseline is defined to the situation where moving average of five data points of 0.15 um particle is lower than 2.0 for a continuous 100 dispenses. 300 mL fresh OK73 is used in both baseline establishment and priming evaluation. After priming completes, a continuous dispense mode is then conducted for 200 dispenses and data from liquid particle counter is collected.

Determination method. A criteria is defined as the following; moving average of five data points of 0.15 μm from the 151st dispense to the 200th dispense is 0.5 count. Recipes that give the value lower than 0.5 is considered pass the criteria and recipes that give the value higher than 0.5 is considered fail the criteria. Ranking of the performance of recipes is conducted by using the average value of 0.15 um calculated from the 151st dispense to the 200th dispense.

RESULT

Table 2. Summary the pass/fail criteria of test result from each recipe

Recipe	Definition of recipe	Pass/Fail criteria
1	Includes soak cycle	Fail
2	Includes backflush to vent cycle	Pass
3	Includes backflush to inlet cycle	Fail
4	Includes filterflush cycle with FP of 2 psi	Pass
5	Includes filterflush cycle with FP of 20 psi	Pass
6	Includes flush bubbles cycle	Pass
7	Includes 10 times repetition of filterflush cycle with FP of 20 psi	Pass

Demonstration of the average value calculated from the 151st cycle to the 200th cycle after priming completes.

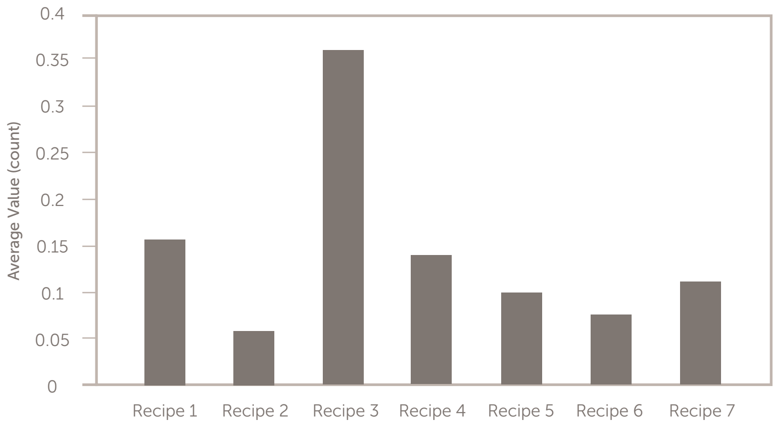


Figure 15. Recipe 2 (includes Soak cycle) showed the best performance, followed by Recipe 6 includes Flush Bubbles cycle), and Recipe 5 (includes FilterFlush cycle), respectively

SETTING OF THE BEST RECIPE (RECIPE 2) ON INTELLIGEN-ULV MMI

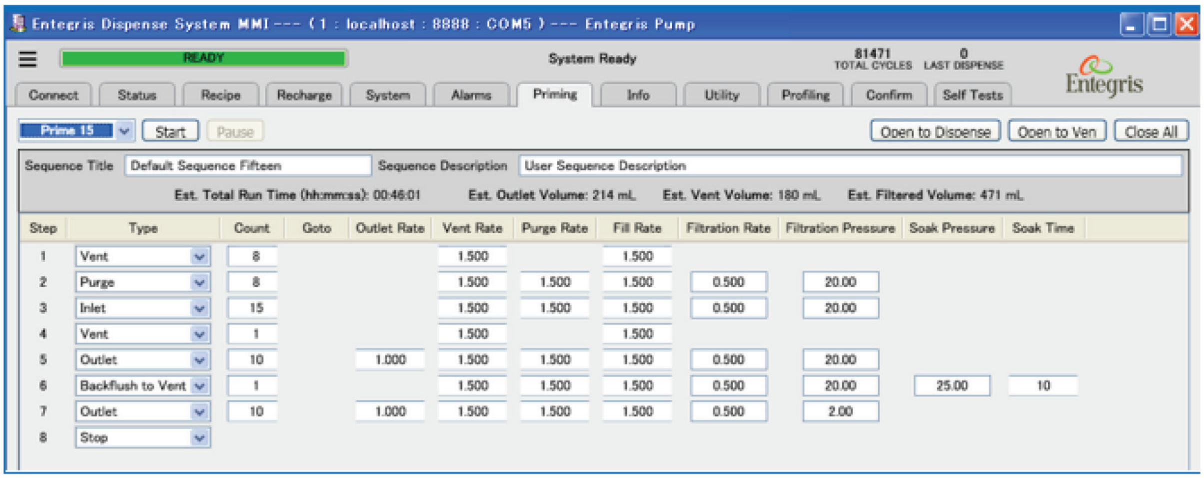


Figure 16. Setting of the best recipe (recipe 2)

SUMMARY

Backflush to vent cycle showed a promising performance. It is the technology that only two-stage dispense system can operate. By filing the membrane pores from the downstream where pore size is tighter, liquid later comes from the upstream can easily flow through membrane pores, facilitating the elimination of small bubbles from filter. Moreover, the IntelliGen ULV features with pressurization system. The pressurization on liquid helps micropores and nanopores can be filled by liquid. Merit of this benefit is not only preventing bubbles released over time but also enhance the filtration efficiency of filter as there are more areas where filtration mechanism can occurs.

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