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Understanding Particle Contribution from Components Used in Ultrapure Water and High-Purity Chemical Systems and Their Impact on Industry-Driven Particle Requirements

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Creative Technology CT Associates, Inc

UPW and Chemical Particle Challenges (IRDS)

Year of Production	*2018*	***2019***	*2020*
Logic industry "Node Range" Labeling (nm)	"10"	**"7"**	"7"
IDM-Foundry node labeling	i10-f7	**i7-f5**	i7-f5
Critical particle size non-electrically active (non-EAP) (nm) based on 50% of Logic 1/2 Pitch (nm)	9	7	7
Ultrapure Water			
PROACTIVE: Non-EAP Particle Control: 50nm (#/L), Feed to the Final Filter	<220	<220	<220
HP materials used in distribution of UPW			
Number of particles >critical particle size (#/l), non-electricaly active particles	**10000**	**10000**	**10000**
Proactive measured using SEMI F104 50nm #particles/l	140	140	80
HP materials used in distribution of Chemicals			
All cleaning chemistries (aqueous and solvent): number of particles /l > critical particle size	**<10**	**<10**	**<10**

Risks?

- Limited ability to detect and measure particles at the critical size
- Limited capability for particle capture at these sizes

Problem Statement

- While industry particle cleanliness requirements have been decreasing rapidly, industry test specifications have not been keeping up with the requirements.

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- In particular, SEMI F104-0312, “Particle Test Method Guide for Evaluation of Components Used in Ultrapure Water and Liquid Chemical Distribution Systems”, does not adequately address the needs of current and projected size requirements for particle contamination.
  - Critical particle diameter is currently 7 nm and going down.
  - 100 nm is the specified particle dimension in the spec.
  - Only specification requirement is for valves.

Problem Statement

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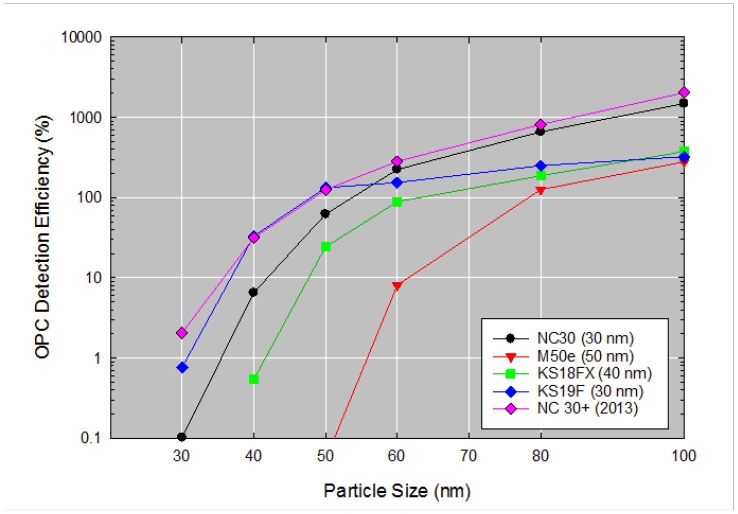
- In particular, SEMI F104-0312, “Particle Test Method Guide for Evaluation of Components Used in Ultrapure Water and Liquid Chemical Distribution Systems”, does not adequately address the needs of current and projected size requirements for particle contamination.
  - Critical particle diameter is currently 10 nm and going down.
  - 100 nm is the specified particle dimension in the spec.
- State of the art optical particle counters (OPC’s) are not capable of measuring the critical particle size.
  - Lowest detection limit available 20-30nm.
  - Low detection particle efficiency (< 5%) at these sizes.
  - Detection efficiency vary between manufacturers and instruments.
  - New technologies such as acoustic emission (Particle Scout) and nebulization and aerosol sizing (Scanning Threshold Particle Counter and Liquid Nanoparticle Sizing) however, correlation to historical OPC data is not well understood.

Low Detection Limit Optical Particle Counters

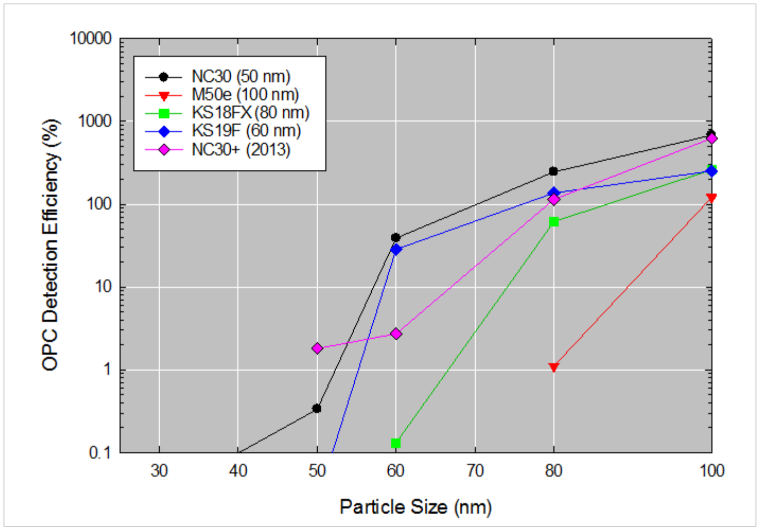
Manufacturer and Model	First Channel (nm)	Second Channel (nm)	Third Channel (nm)	Fourth Channel (nm)	False Counts (#/mL)
Lighthouse NC30+	30	50	80	100	0.02
Rion KS-19F	30	60*	100*	130*	0.10
PMS Ultra DI®20	20	50	70	100	0.05

* Rion channel size is selectable

First Channel Detection Efficiency



Second Channel Detection Efficiency



Source: Van Schooneveld, et al., “Counting efficiency comparison of liquid optical particle counters below 100 nm”, UPW Micro 2015

SEMI F104 Rewrite Technical Approach

- Use 7 nm as the critical particle size objective.
- Use OPC's as the principle measurement technology.
- Use a power law analysis ($f(x)$=k∗(1/d^n) to establish an appropriate specification in the range of the OPC's where:
    - k = Cumulative number concentration (#/mL) $\geq$ d (nm)
    - d = channel size (nm)
    - n = slope exponent (typically ranges from 2.5 to 3.5)
- Validate selected methodology using a variety of components (valves, tubing and pressure regulators).
- Develop fluid system distribution models and establish particle budgets to critical fluid components.
- Evaluate new 10nm instruments such as scanning TPC to begin establishing correlation between the measurement technologies.

Collaboration Team

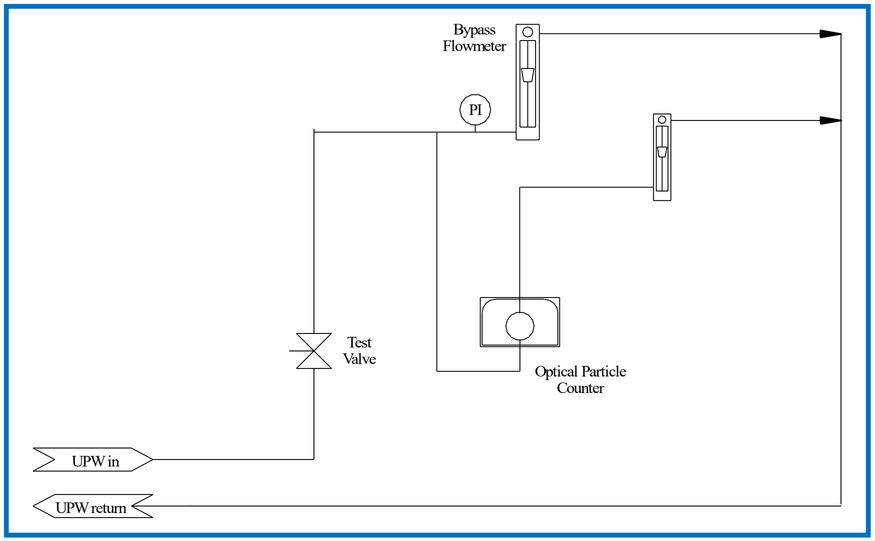
- Seven material and component suppliers:
  - Altaflo
  - Asahi/America
  - Entegris
  - GEMU
  - Georg Fischer
  - Parker Hannifin
  - Saint Gobain Performance Plastics
- 1 OPC manufacturer - Lighthouse Worldwide Solutions
- Entegris – Rion KS-19F OPC Loaner
- All testing conducted by CT Associates (Eden Prairie, MN)

Test Matrix

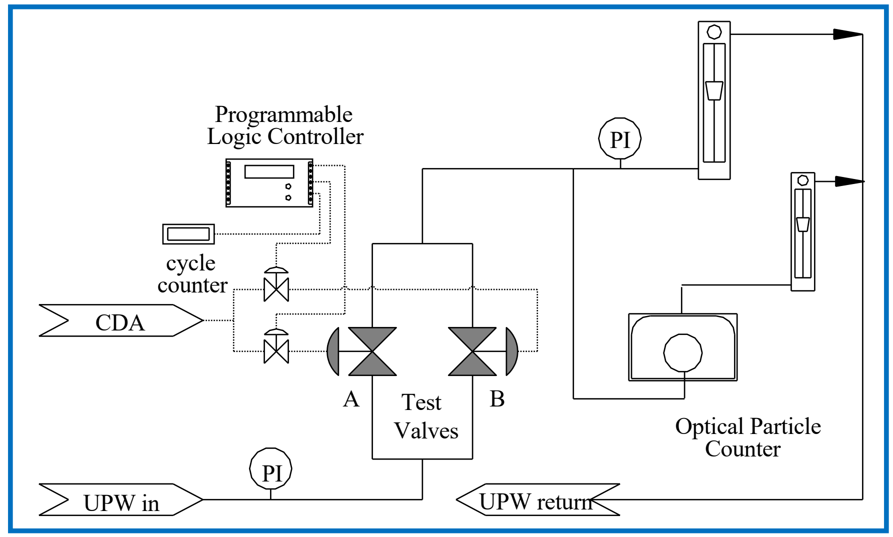
- 5 sets (4 each) – ½” air-operated valves
- 1 set (4 each) – 32 mm air-operated valves
- 1 set (2 each) – 63 mm air-operated valves
- 3 sets (4 each) – pressure regulating devices
- 8 - 15 meter lengths – PFA* tubing

* Two PFA raw material suppliers were used in the extrusions

Test Apparatus – Valves and Tubing

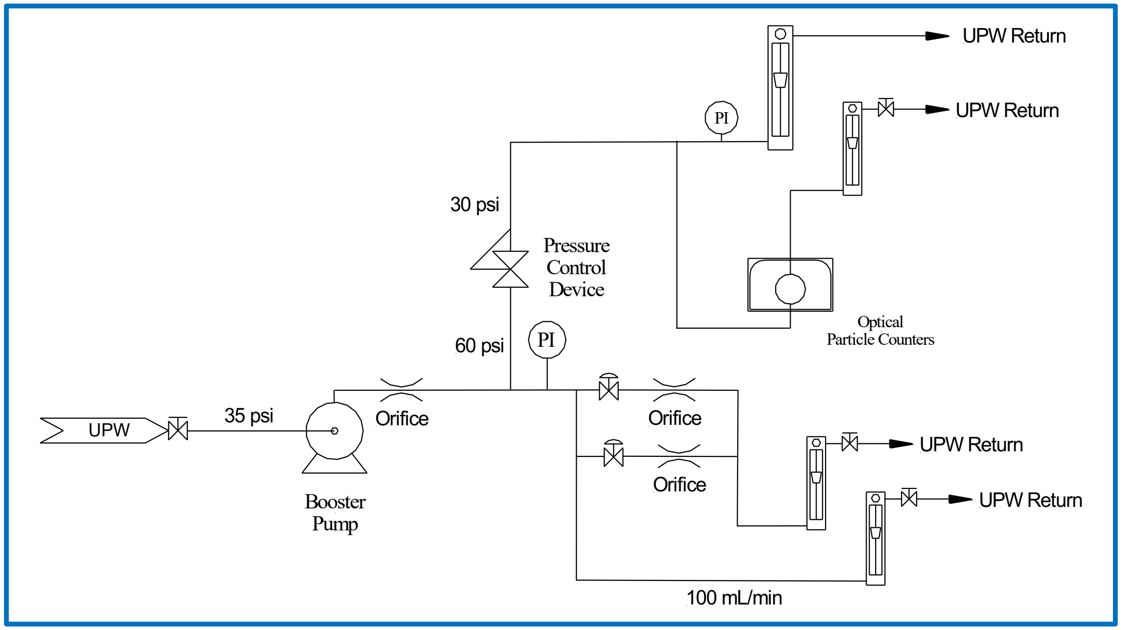


Valve Static (No Actuation) Rinse

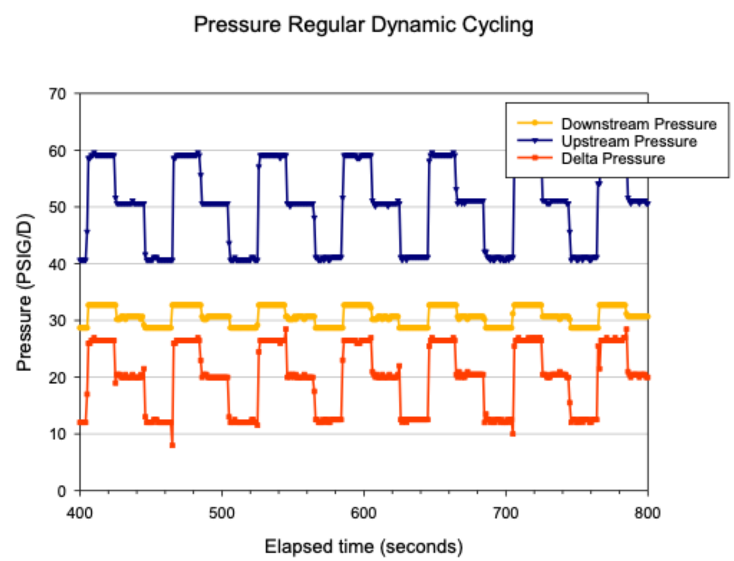


Valve Dynamic (Valve Cycling) Rinse

Test Apparatus – Pressure Control Devices

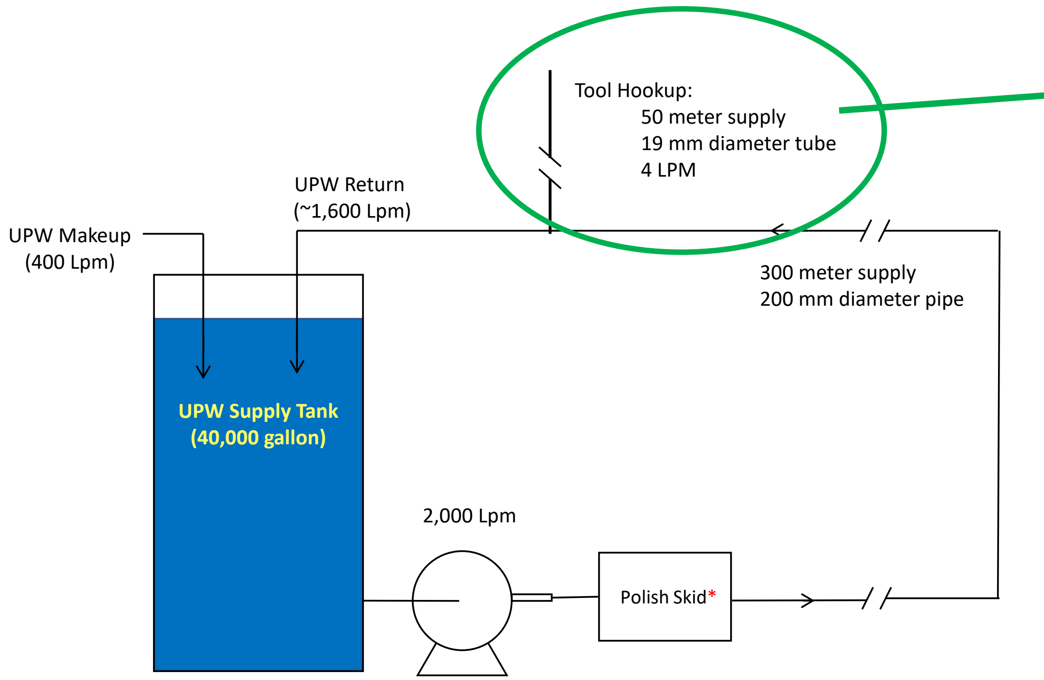


Pressure Control Device - Dynamic Inlet Testing



Complete Cycle – 30 seconds

Distribution Model – Mains and Hook-ups



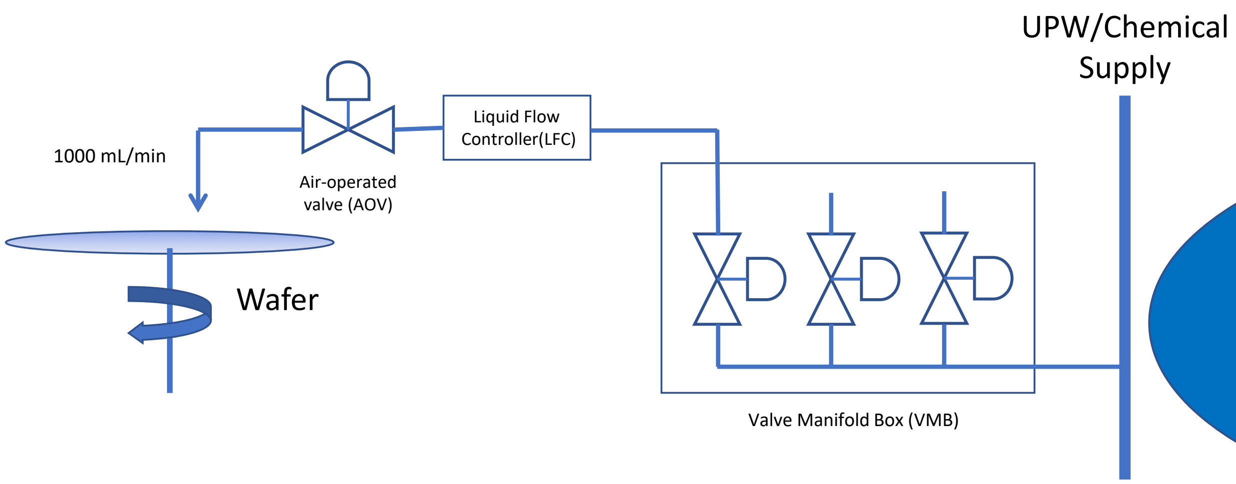
Killer Particle Size:	7	nm		
Killer Particle Concentration:	51	#/mL	Proactive IRDS Roadmap	
Total Particle Budget:	25	%		
Power factor exponent:	3			
Tubing Budget:	100	%		
Concentration budget at killer particle size	12.8	#/ml		
Total particle allowable at:	20	nm	0.547	#/ml
	30	#/ml	0.162	#/ml
	40	#/ml	0.068	#/ml
	50	#/ml	0.035	#/ml
	100	#/ml	0.004	#/ml
Tubing Allowable assuming 50 meter run:	20	nm	0.109	#/ml/10 meter
	30	nm	0.032	#/ml/10 meter
	40	nm	0.014	#/ml/10 meter
	50	nm	0.007	#/ml/10 meter
	100	nm	0.001	#/ml/10 meter

Particle Budget = 50%
Killer particle dimension = 7 nm
PSD: $f(x) = k * (1/d^n)$ where n=3
Particle Allocation – Main 25%, Hook-up 25%

≥ 50 nm Tubing Particle Budget:

0.007 particles added/mL/10 meters

Point of Use Model – Valves and Flow Controllers



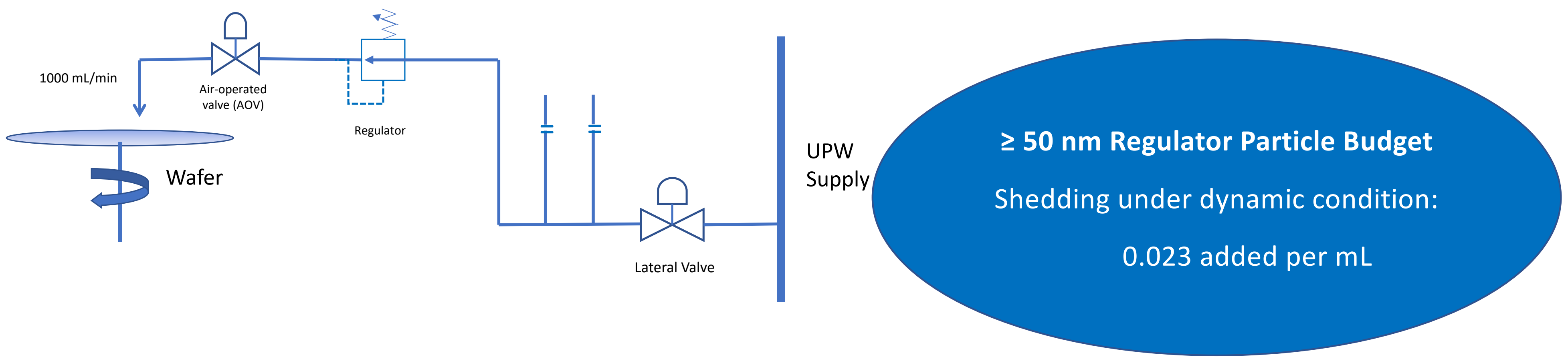
≥ 50 nm Valve and FC Particle Budget:

Rinse: 0.002 added per mL

Generation: 0.021 added per mL (or 21 particles per actuation at 1,000 mL/min)

POU Particle Budget = 50%
Killer particle dimension = 7 nm
PSD: $f(x) = k * (1/d^n)$ where n=3
Particle Allocation = 10% steady state shedding + 90% generation from actuation

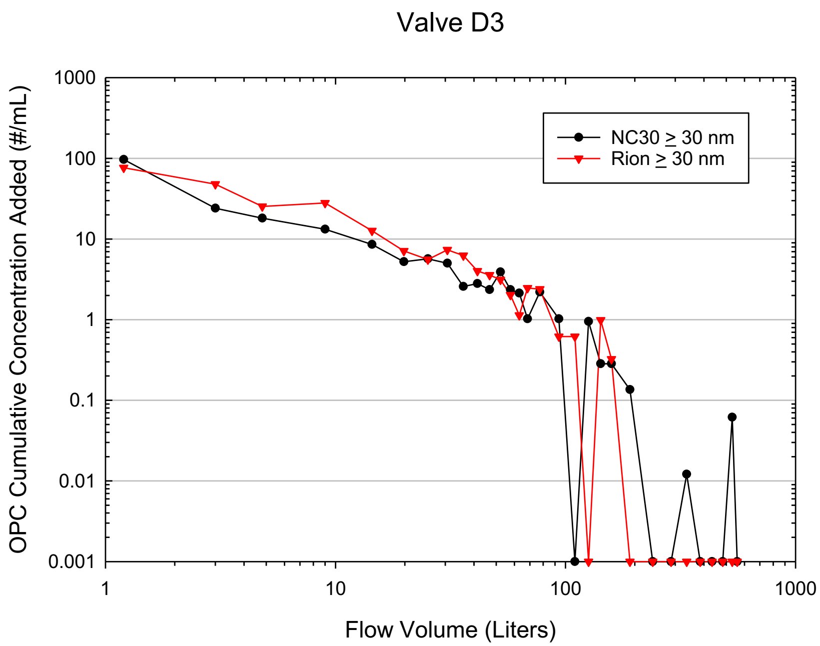
Point of Use Model –Regulators

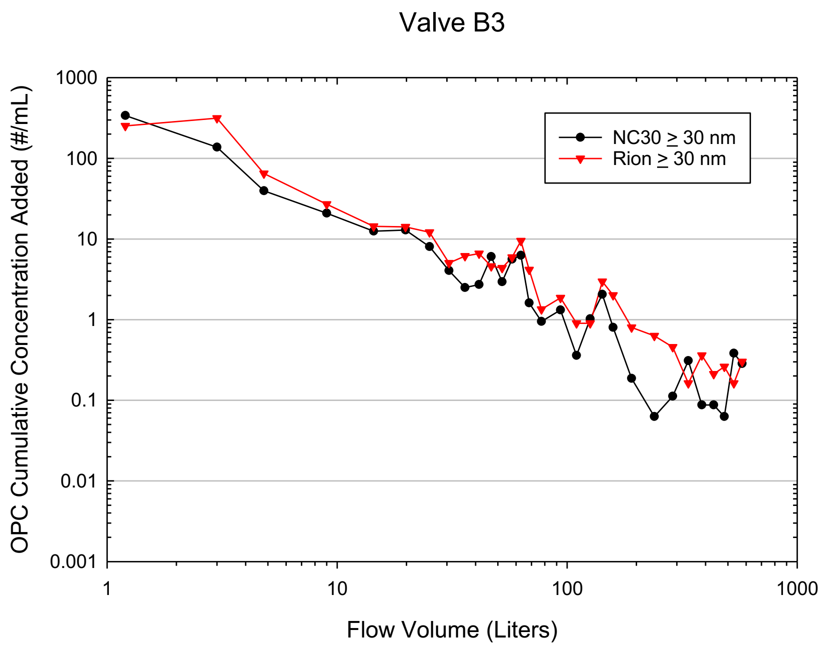


Particle Budget = 50%
Killer particle dimension = 7 nm
PSD: $f(x) = k * (1/d^n)$ where n=3
Particle Allocation (Regulator) = 100% Dynamic Steady State

Test Results

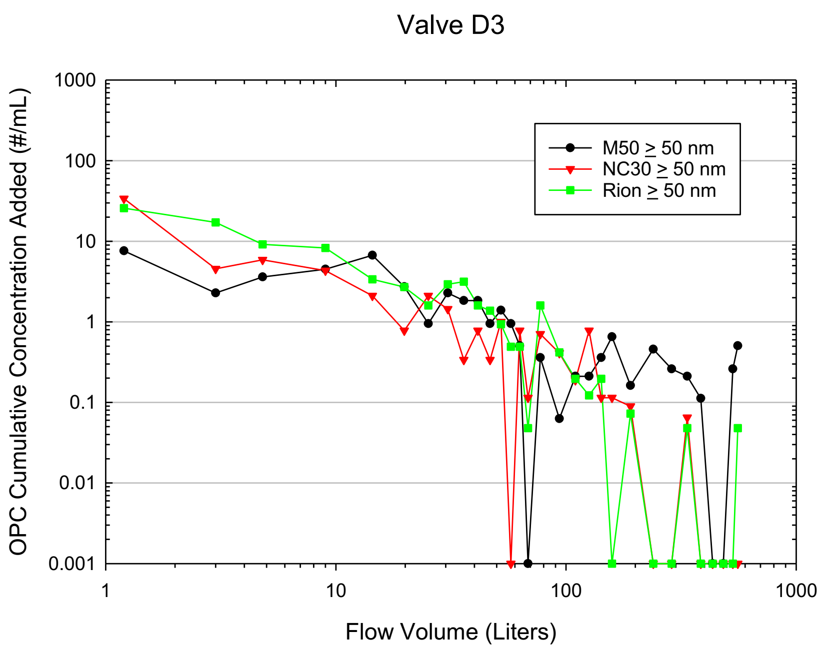
OPC Comparisons - ½” AOV Rinse Results - 30 nm OPC Channel

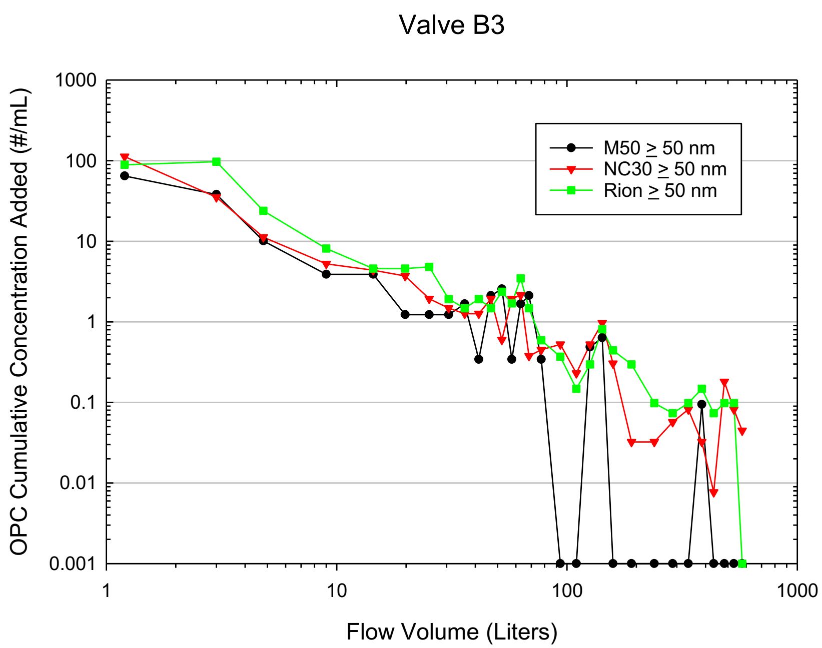




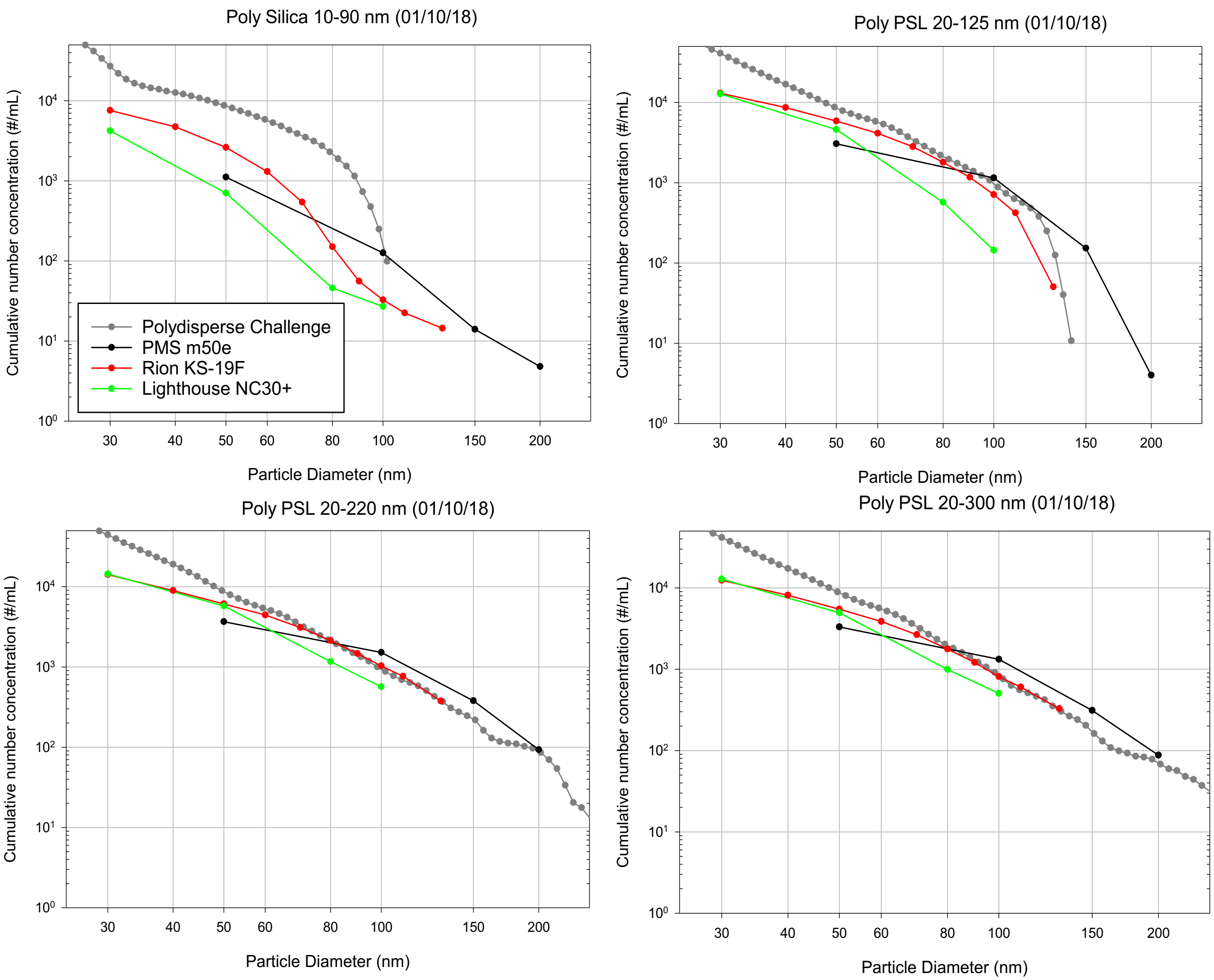
Both 30 nm OPC provided similar 30 nm results during the rinse testing.

OPC Comparisons - ½" AOV Rinse Results - 50 nm OPC Channel



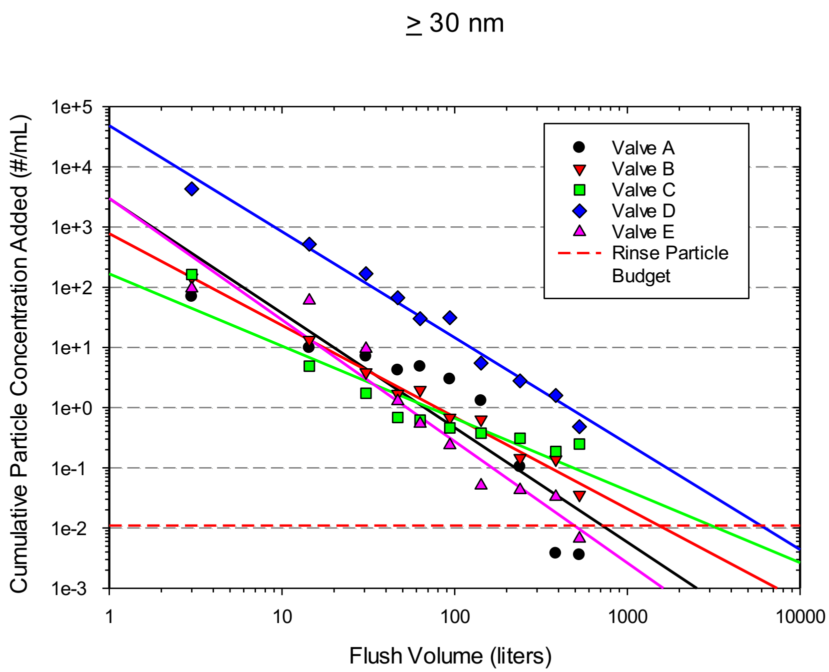


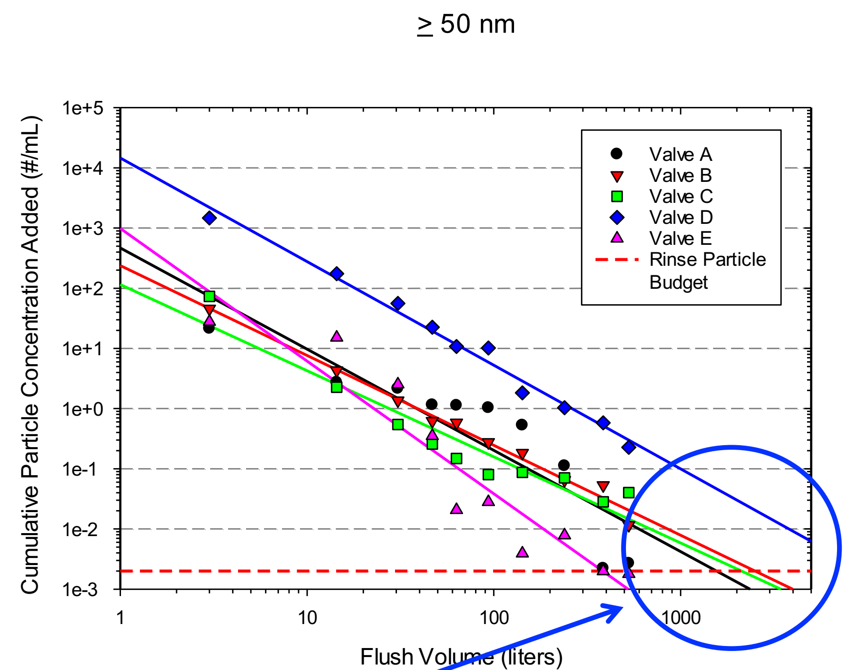
All three OPC provided very similar 50 nm channel results during the rinse testing.



PSL testing supported the correlation observed between the OPCs during the rinse tests.

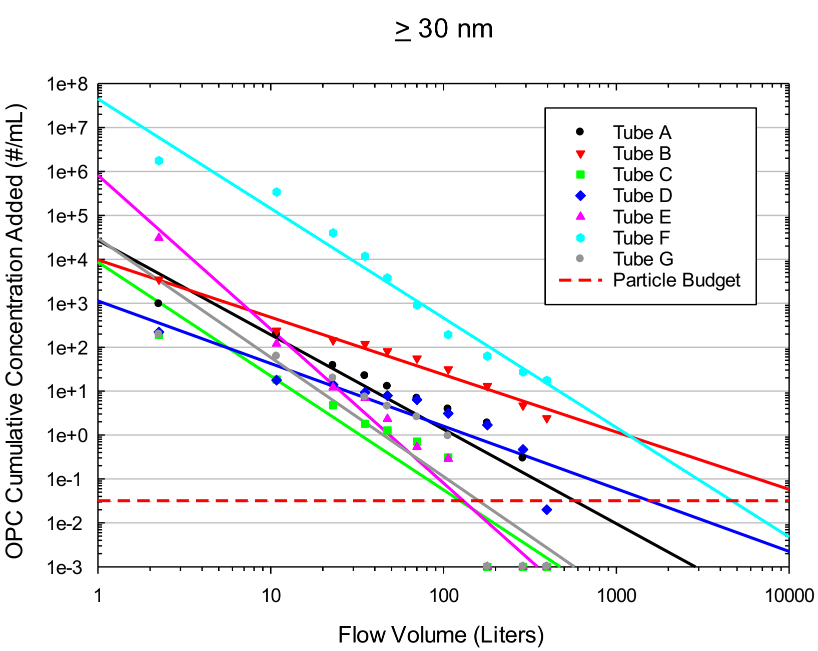
Valve Rinse Test Summary – 30 and 50 nm

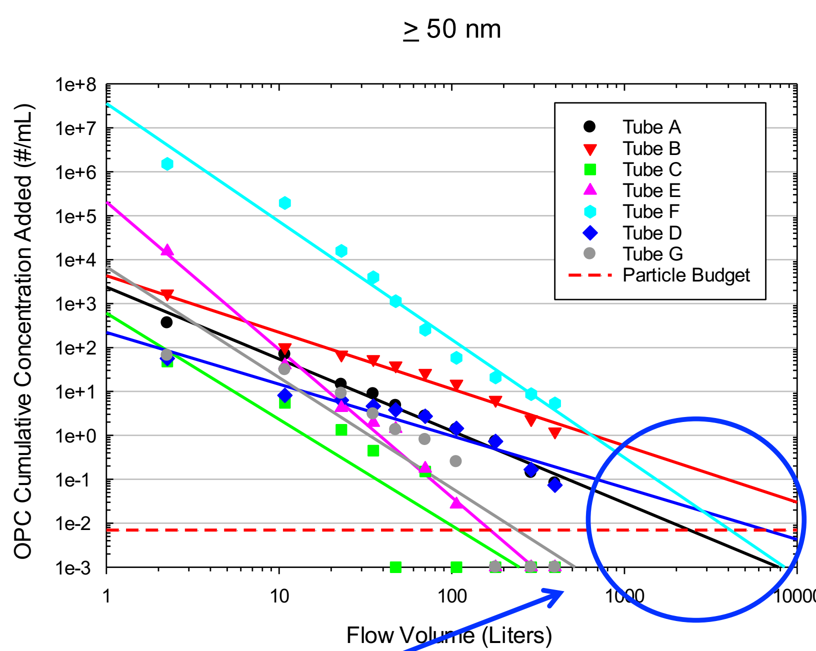




~ 30 hours rinse time at 1 LPM to reach 0.002 particles added ≥ 50 nm

½” Tubing Rinse Test Summary – 30 and 50 nm

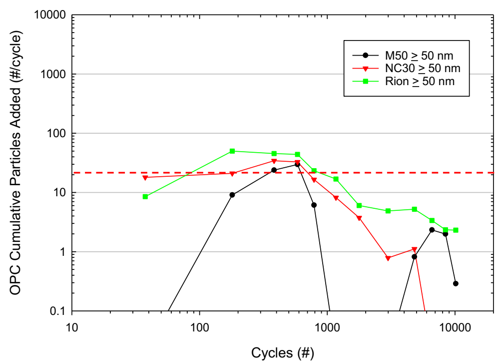




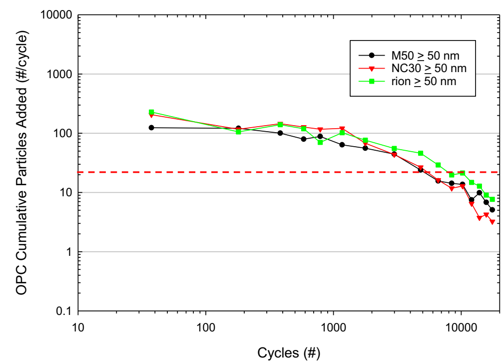
~ 24 hours rinse time at 4 LPM to reach 0.007 particles added ≥ 50 nm

Valve Cycling – 50 nm

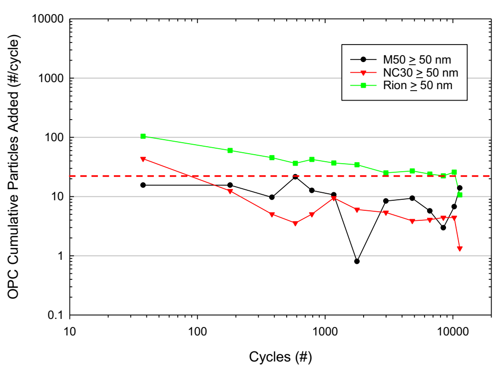
Valve A1/A2



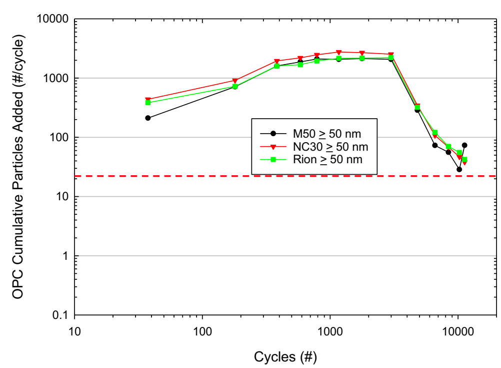
Valve B1/2



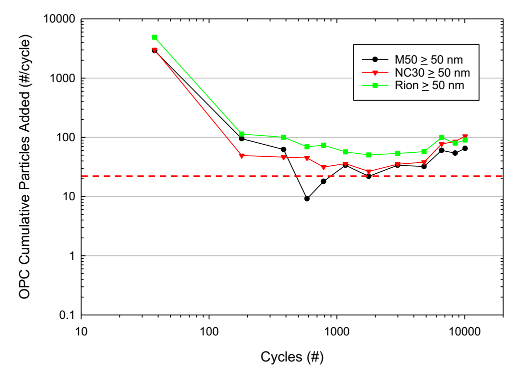
Valves C1/2



Valves D1/2

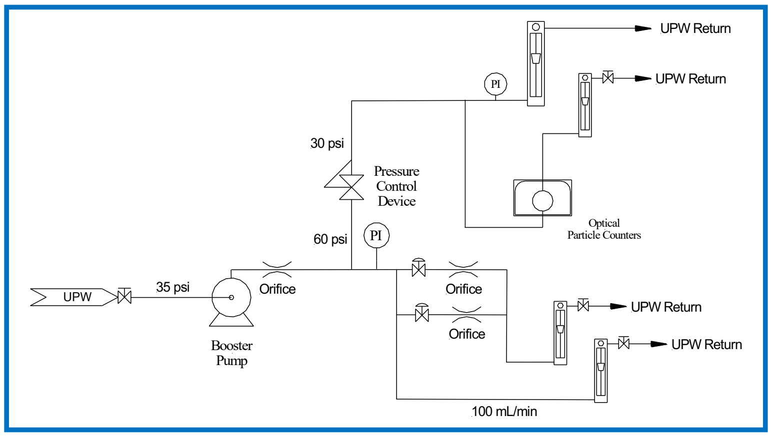


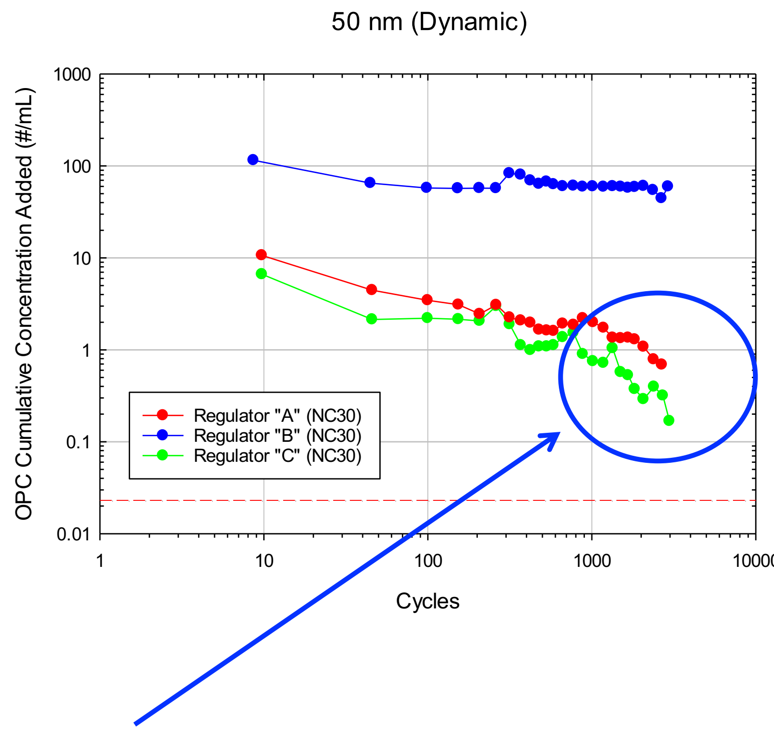
Valve E1/2



Three of the five valves would meet the shedding model requirement of 21 particle per actuation (~ 0.021/mL at 1000 mL per minute) after sufficient cycling .

Regulators under Dynamic Inlet Conditions – 50 nm





Eventually, two of the three regulators tested would likely meet the rinse model requirement of 0.023 particles per mL added but with extensive cycling and added flow volume.

Planned Changes to the SEMI F104 Specification

- Particle specification will move from SEMI F57 to SEMI F104.
- Critical OPC channel size will reduce from 100 nm to 50 nm.
- Specification will be expanded to include flow controllers and flow-through components such as flowmeters, pressure sensors and tubing.
- Particle rinse specification will be based on rinse volume and vary as a function of component size (i.e. flush volume spec will increase as the component fluid-path cross-section increases).
- Testing of pressure control devices under dynamic inlet conditions will be a requirement but no performance specification will be established at this time, only that results are reported.

Summary

- All valves tested in this study were able to meet the IRDS pro-active particle requirement budget as modeled during static rinse.
- The majority of the valves tested were able to meet the IRDS pro-active particle requirement budget as modeled under dynamic (cycling) conditions.
- While the majority of the valves tested were able to meet the model requirements, there was considerable variability in particle shedding and/or generation performance and most exhibited a “break-in” period to meet the target. This is an area for potential improvements opportunities.
- All tubing tested in this study were able to meet the IRDS pro-active particle requirement budget as modeled.
- The pressure control devices tested in this study under dynamic inlet conditions were challenged to meet the IRDS pro-active particle requirement budget.

Acknowledgements

- Bob McIntosh, Enviro-Energy Solutions for leading the SEMI F104 effort.
- The companies providing test components and financial support:

Altaflo	Asahi/America
Entegris	GEMU
Georg Fischer	Parker Hannifin
Saint Gobain	

- Entegris for loaning one of their Rion KS-19F for part of the study.
- Lighthouse Worldwide Solutions – NC30+ calibration

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