Counting efficiency comparison of liquid optical particle counters below 100 nm

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Introduction

- Optical particle counters (OPCs) have broad application within the semiconductor industry ranging from UPW and chemical monitoring, contamination troubleshooting and component testing (e.g. valves, filters, pumps, containers, etc.).

- Component research, development, testing and qualification may occur at many locations within a company or involve multiple companies.

- Comparing OPC data generated by multiple counters can be challenging as OPCs and their associated supply systems may have differing background levels and detection efficiencies depending on issues such as system cleanliness, laser status, time since calibration or changes caused by transportation and handling.

- The ability to characterize counting efficiency becomes important when trying to determine if differences in OPC data are significant or a result of OPC variability.

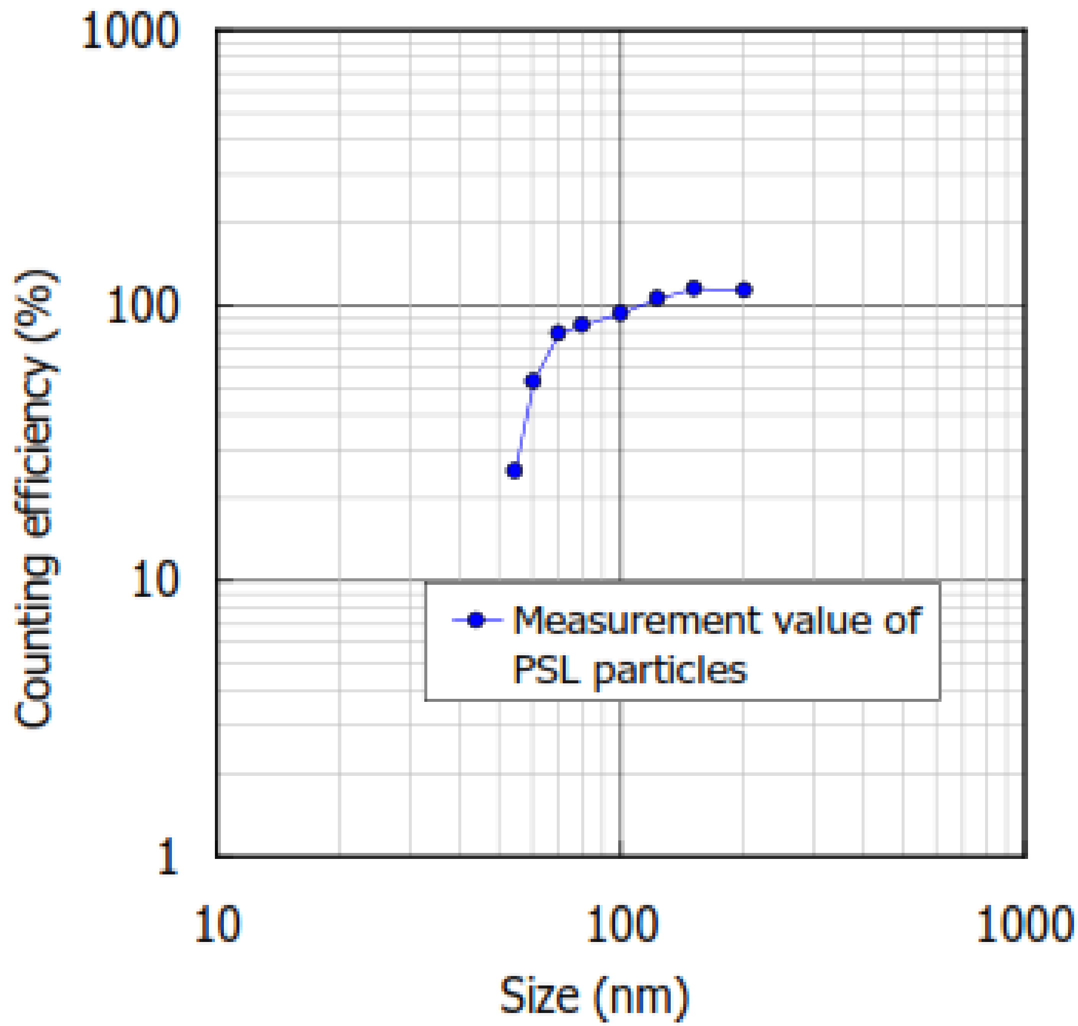
- This paper will examine the use of polystyrene latex (PSL) size and concentration standards to determine the detection efficiency of several commercially available OPCs.

- This paper will also examine the response of several commercially available OPCs to colloidal silica standards.

Outline

- SEMI C77-0912, Method for Determining the Counting Efficiency of Liquid-borne Particle Counters for which the Minimum Detectable Particle Size is between 30 and 100 nm.
- Polystyrene latex (PSL) particle size distributions (PSDs).
- Colloidal silica PSD.
- Test details.
  - Procedures
  - Instrumentation
- Test results.
- Summary

SEMI C77-0912 Overview

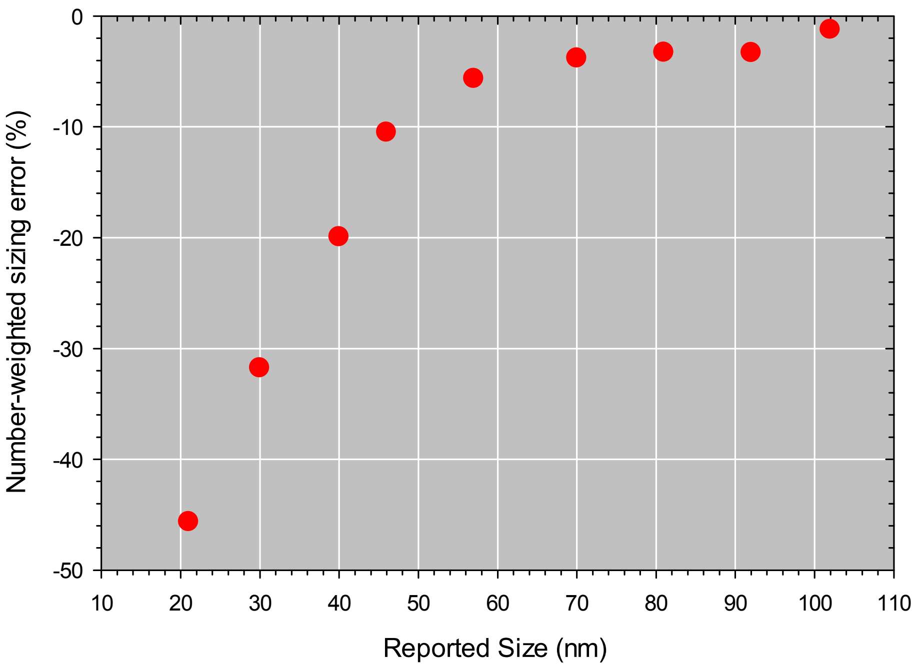


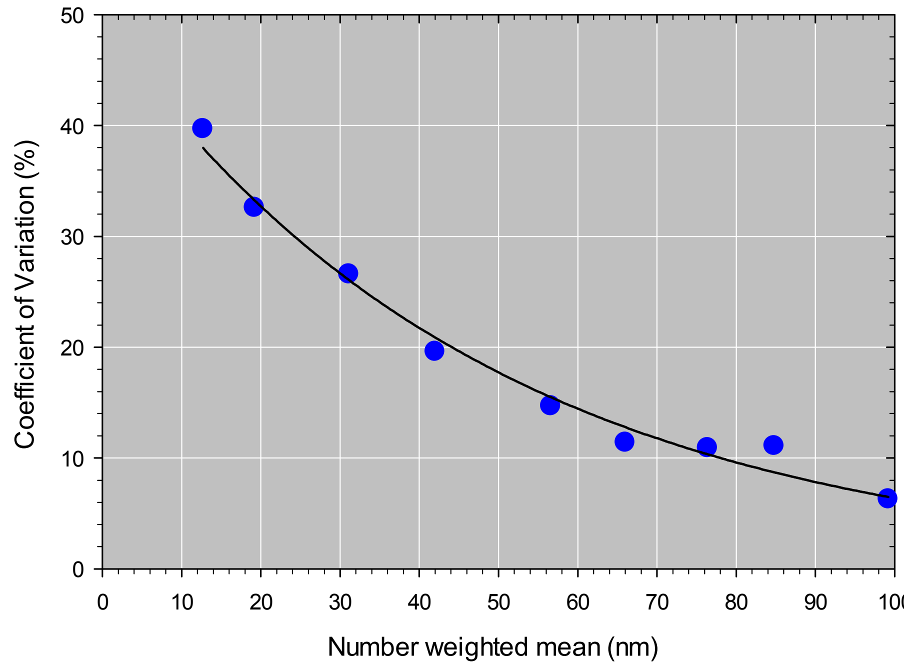
Source: SEMI C77-0912

Impact of PSL PSD on SEMI C77

1. The manufacturer's mean size is typically a volumetric-weighted mean determined by DLS. Concentration standards should to be based on the number-weighted mean. These two means can deviate.

2. Coefficient of variation (standard deviation divided by the mean) increases as the particle size gets smaller.





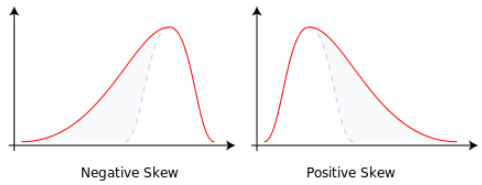
Implication: Up to 30% concentration error as 30 nm is approached.

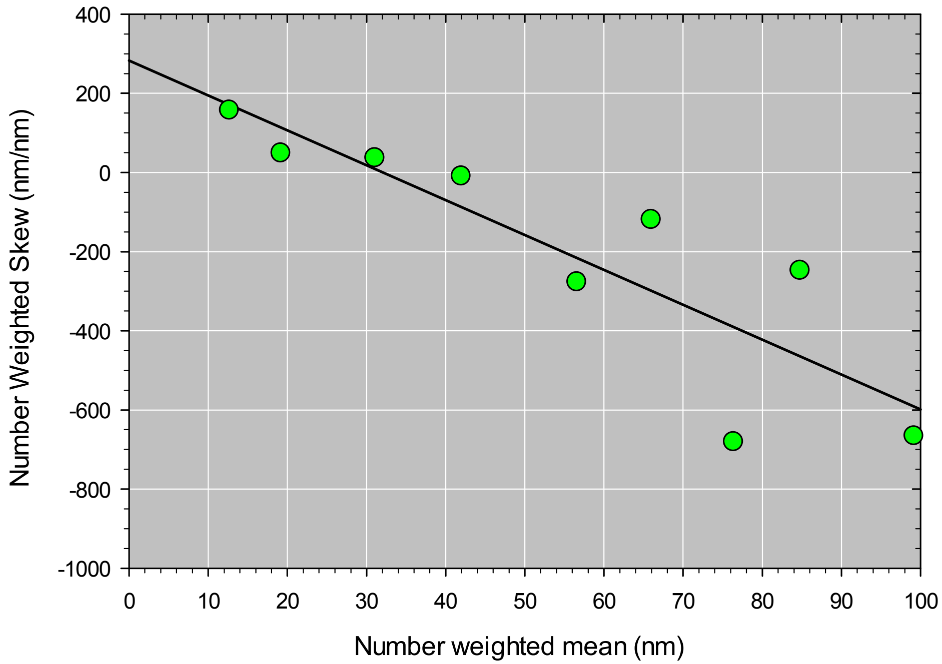
Implication: Up to 25% concentration error as 30 nm is approached.

Reference: Van Schooneveld, et al. "Validating the counting efficiency of liquid optical particle counters below 100 nm", Ultrapure Water Micro 2013, November 12-13, Portland, OR

Impact of PSL PSD on SEMI C77

3. PSL PSD are typical skewed, not normally distributed smaller than 100 nm.





Implication: Up to 15% concentration error .

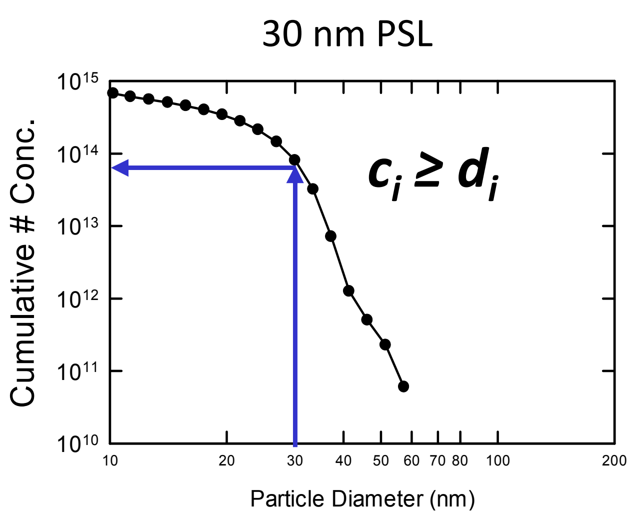
4. Concentration is provided as % solid but there is no accuracy or tolerance data provided.

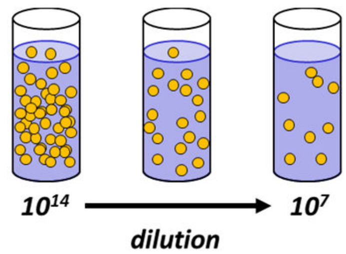
Implication: Up to 30% concentration error has been measured compared to calculating the number concentration from the specified percent solids of 1%.

Key take-away - A well characterized PSL particle size distribution is necessary when determining OPC detection efficiencies below 100 nm.

Reference: Van Schooneveld, et al. “Validating the counting efficiency of liquid optical particle counters below 100 nm”, Ultrapure Water Micro 2013, November 12-13, Portland, OR

Determination of Counting Efficiency



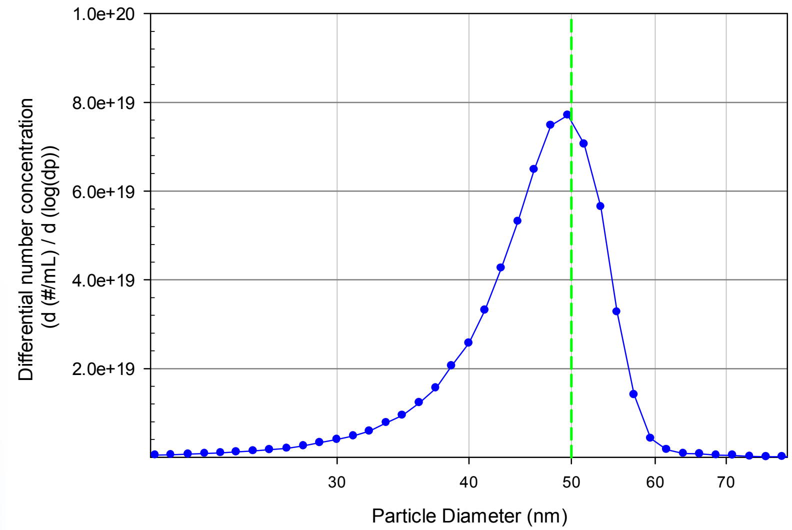


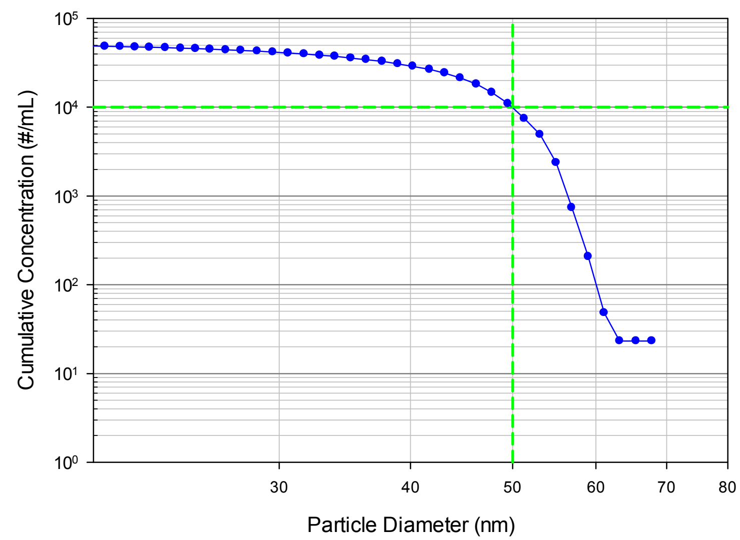
KanomaxFMT **LiquiTrak®**
Precision Dilution System
Model 6138

$$CE\% = \frac{\textit{concentration measured}}{\textit{concentration expected}}$$

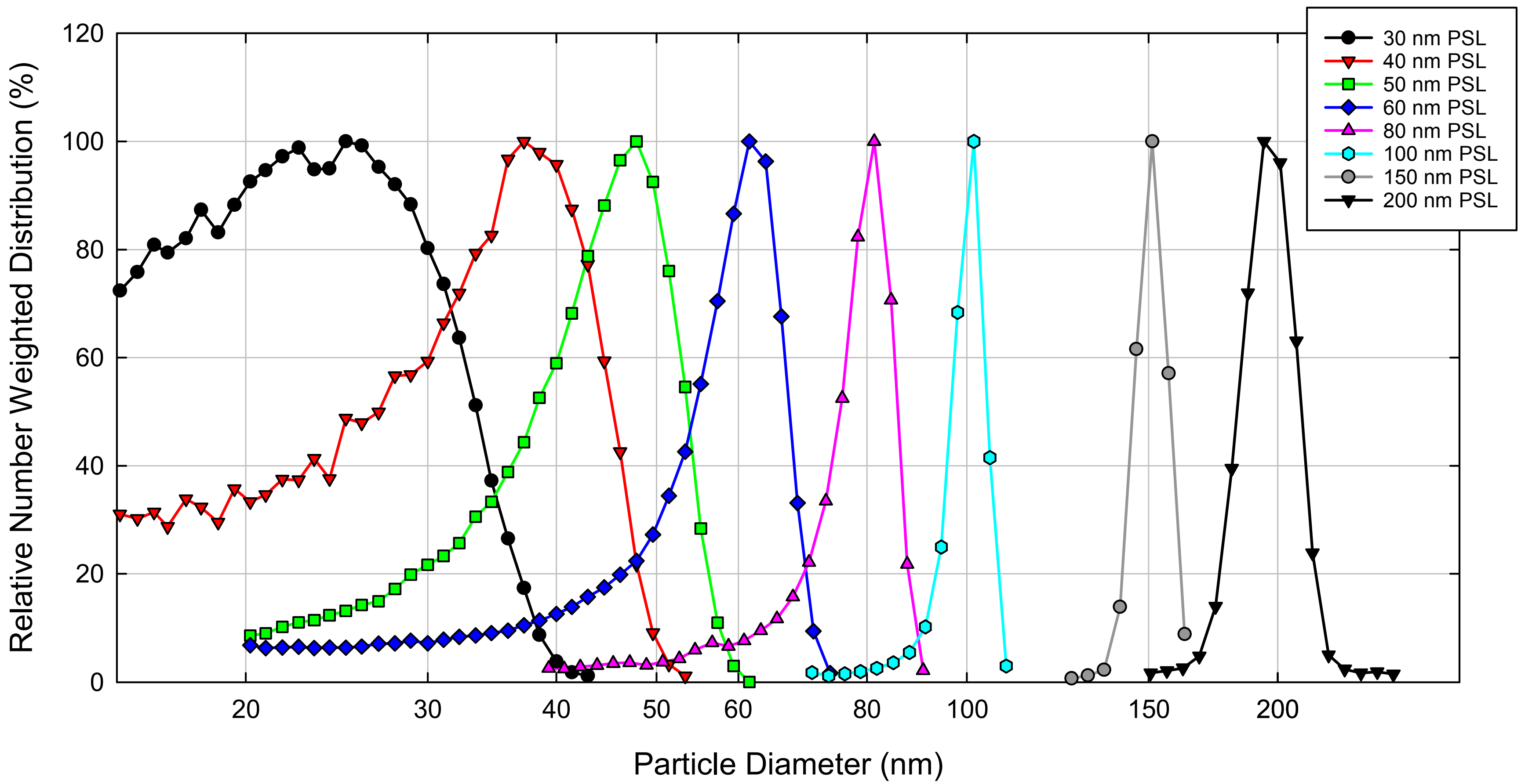
Liquid Nanoparticle Sizing System

Courtesy of Kanomax Fluid Measurement Technologies

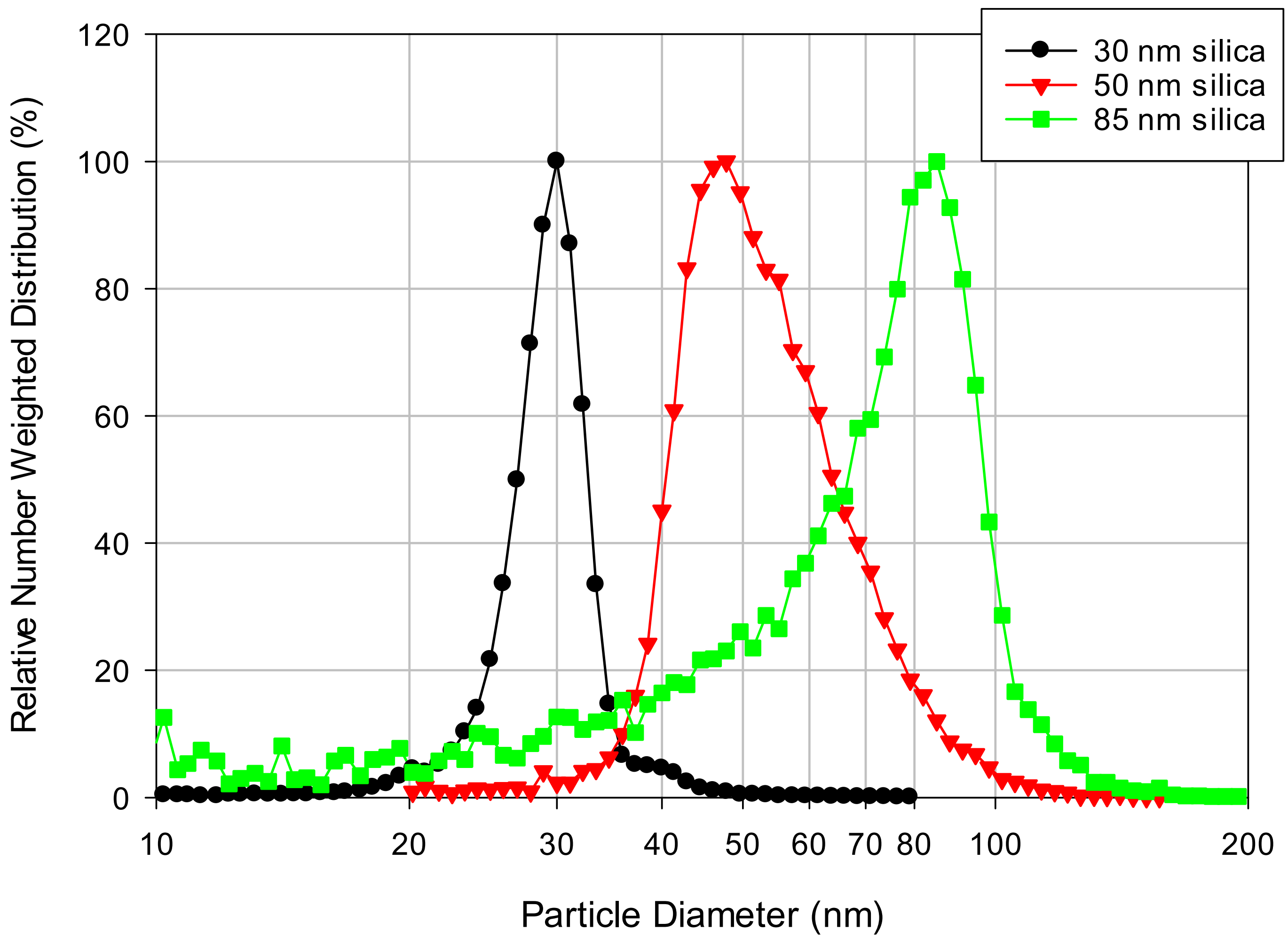




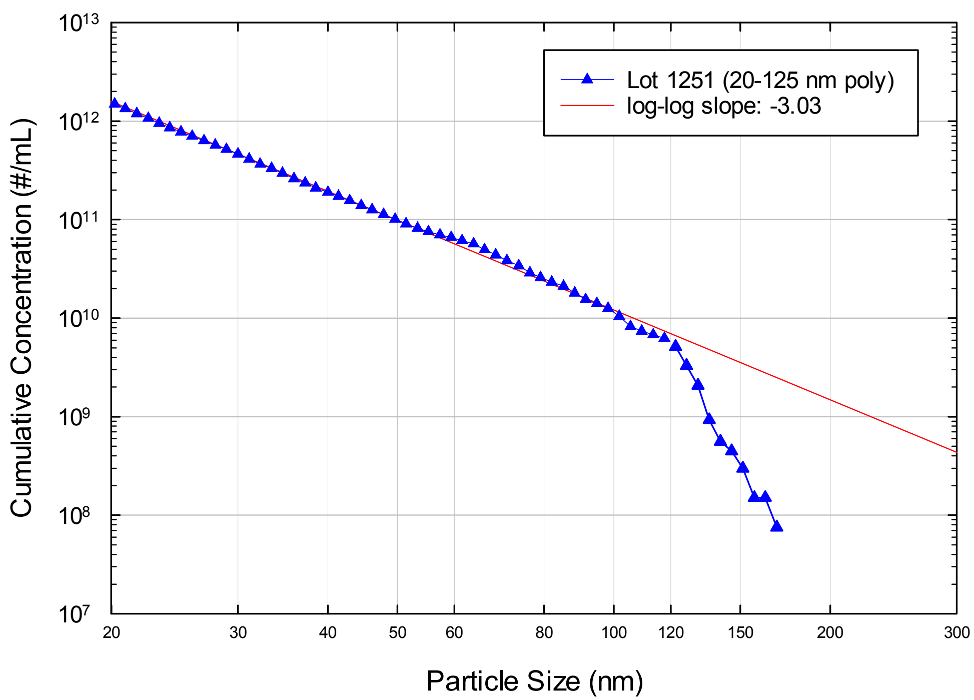
PSL Particle Size Distributions

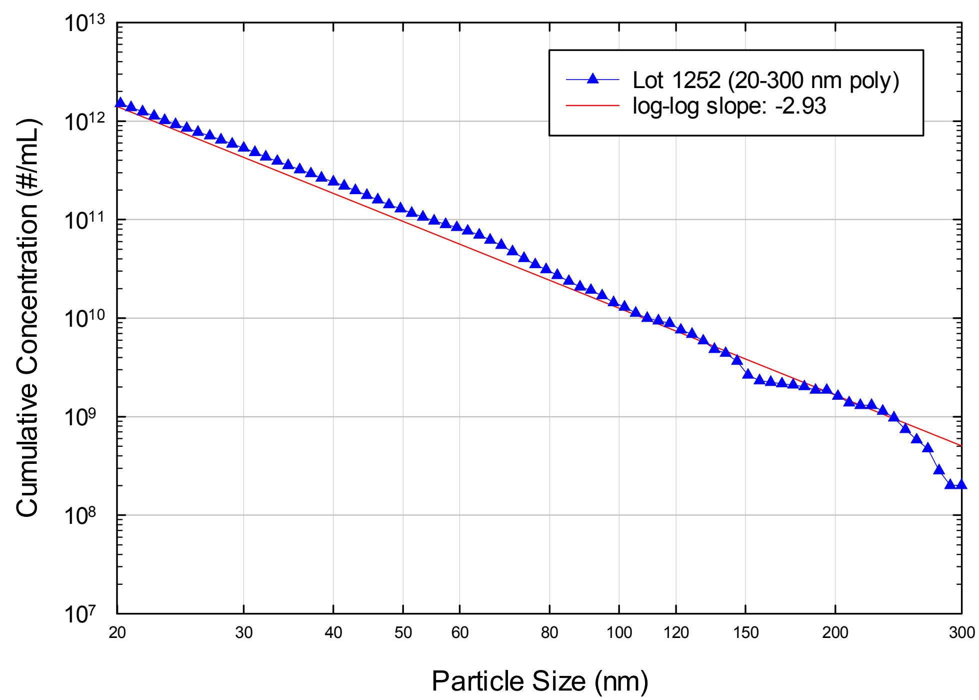


Silica Particle Size Distribution

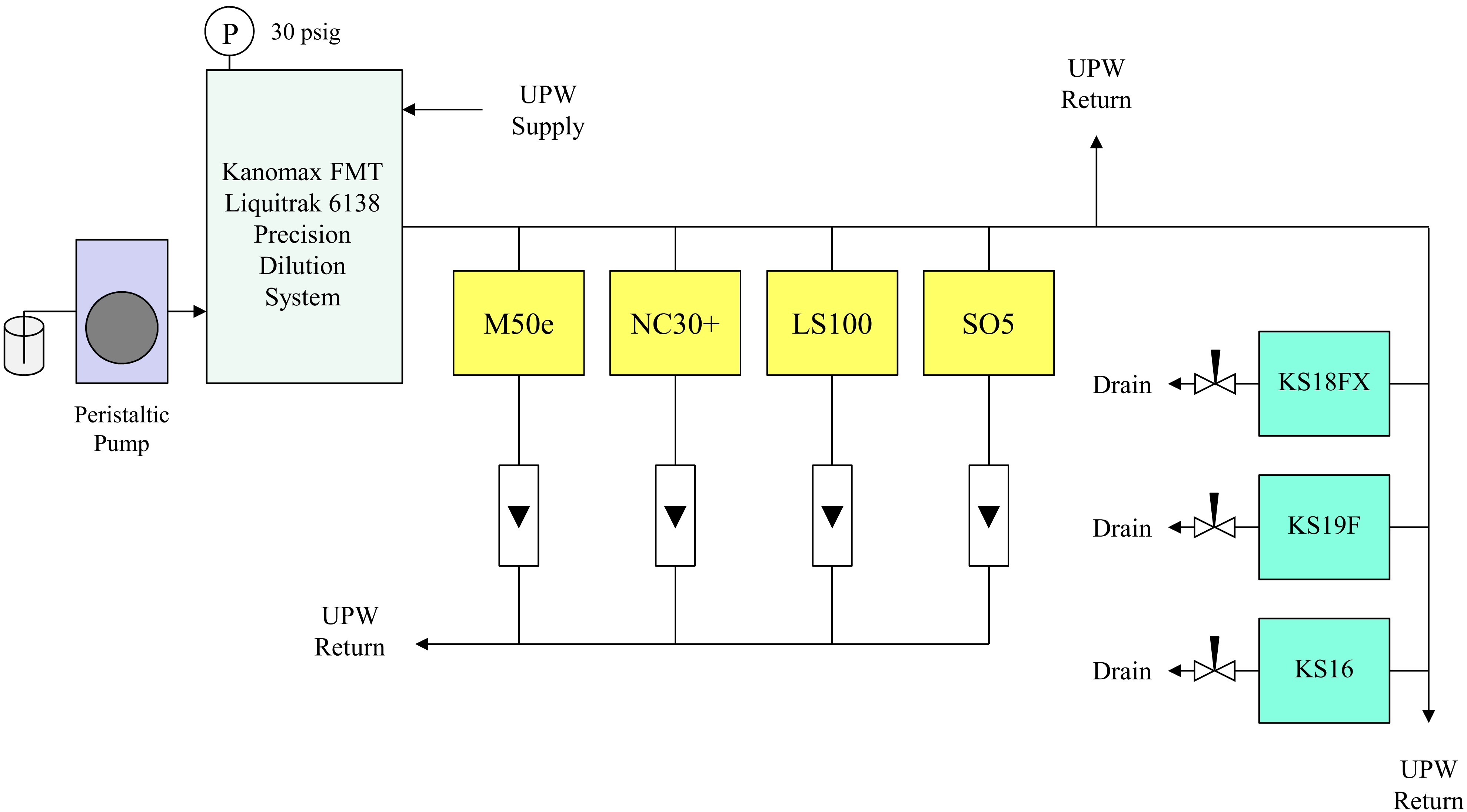


PSL Polydispersed Concentrates





Apparatus



P
30 psig
UPW
Supply
UPW
Return
Kanomax FMT
Liquitrak 6138
Precision
Dilution
System
Peristaltic
Pump
M50e
NC30+
LS100
SO5
Drain
KS18FX
Drain
KS19F
Drain
KS16
UPW
Return
UPW
Return

OPC Details

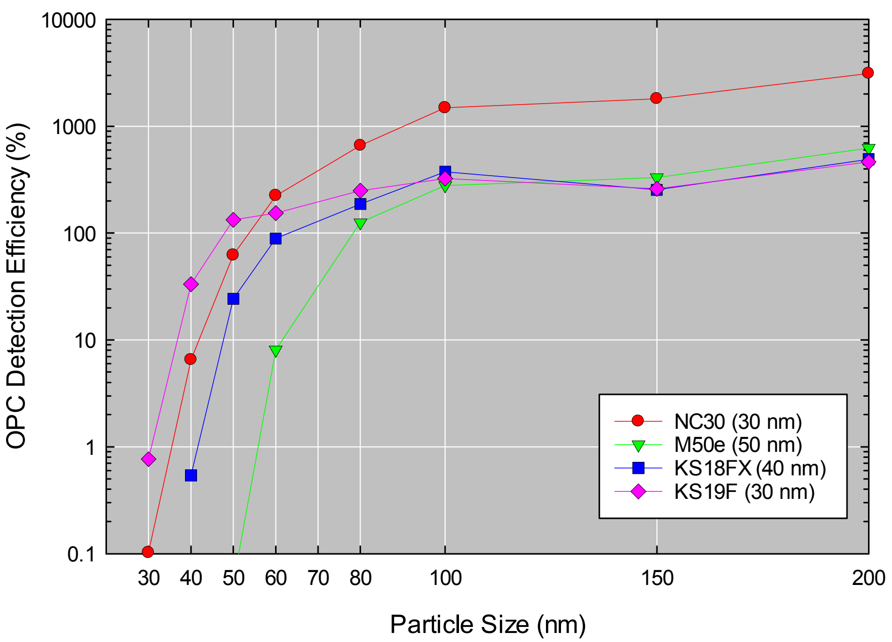
Manufacturer	Model	Serial Number	Calibration Date	Coincidence Limit (p/mL)
Particle Measuring Systems	HS-LIS M50e	35008-436-492	7/22/2015	50,000
	LiquiStat	39288-0699-18	10/15/2012	5,000
	LiQuilaz S05	38681-0199-135/D6	4/15/2012	2,000
RION Co., Ltd.	KS-16	00870398	12/4/2014	1,200
	KS-18FX	00730030	7/31/2015	30,000
	KS-19F	00350037	4/20/2015	40,000
Lighthouse Worldwide Systems	NanoCount 30+	120899001	9/16/2014	1,000,000

Test Procedure

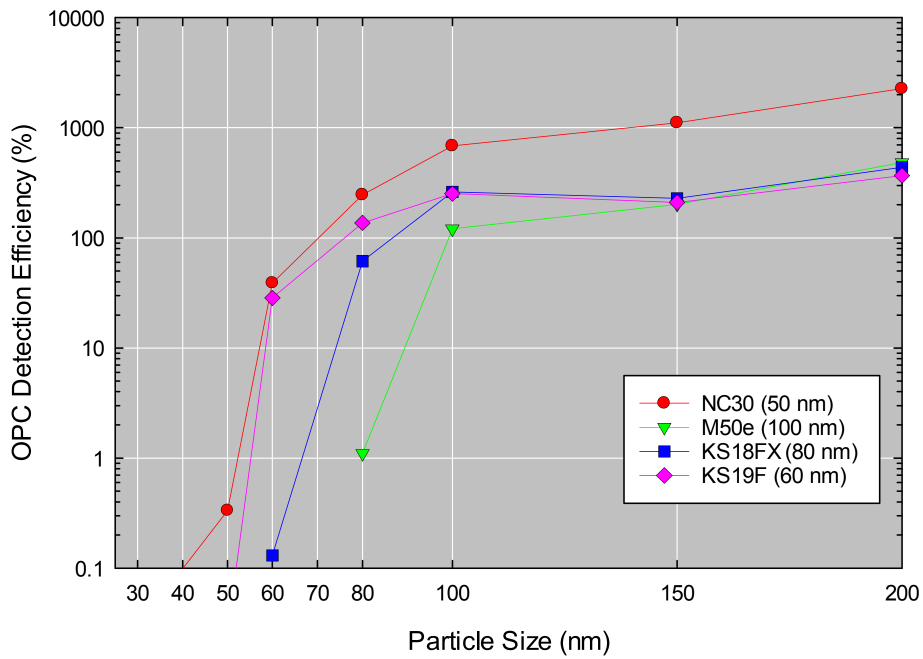
- Prepare PSL and silica concentration standards in UPW.
- Establish dilution ratio (injection rate/dilution flow) based on the coincidence level of the counters tested.
- Set pressure at the particle counter to 15+ psig.
- Ensure OPCs reading at background level.
- Inject particle standard until steady counts are observed for 15 minutes.
- Reverse pump for 5 minutes before injecting next standard.
- Data averaged over last 10 minutes of sampling.

PSL Counting Efficiency Results

First Channel Detection Efficiency



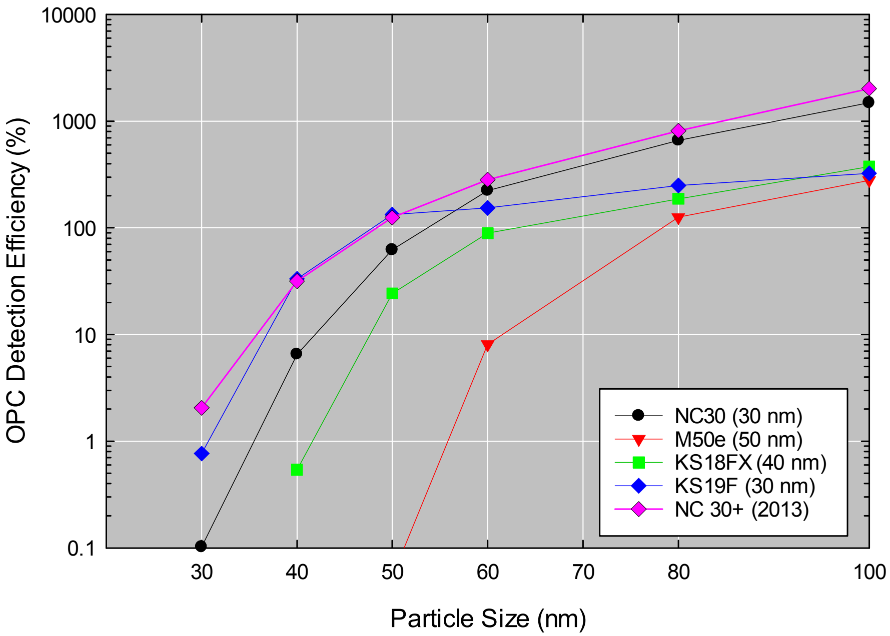
Second Channel Detection Efficiency



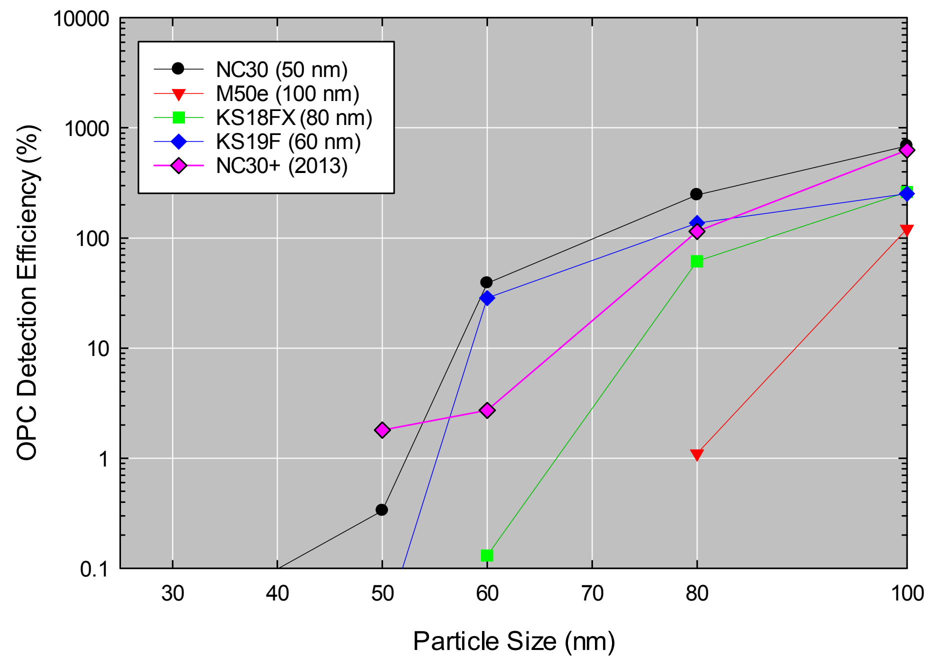
- Significant counting efficiency differences were observed below 100 nm.
- Assuming that the OPCs respond similar to naturally occurring particles, having OPC detection efficiencies allows for data from various counters to be correlated.

PSL Counting Efficiency Changes

First Channel Detection Efficiency

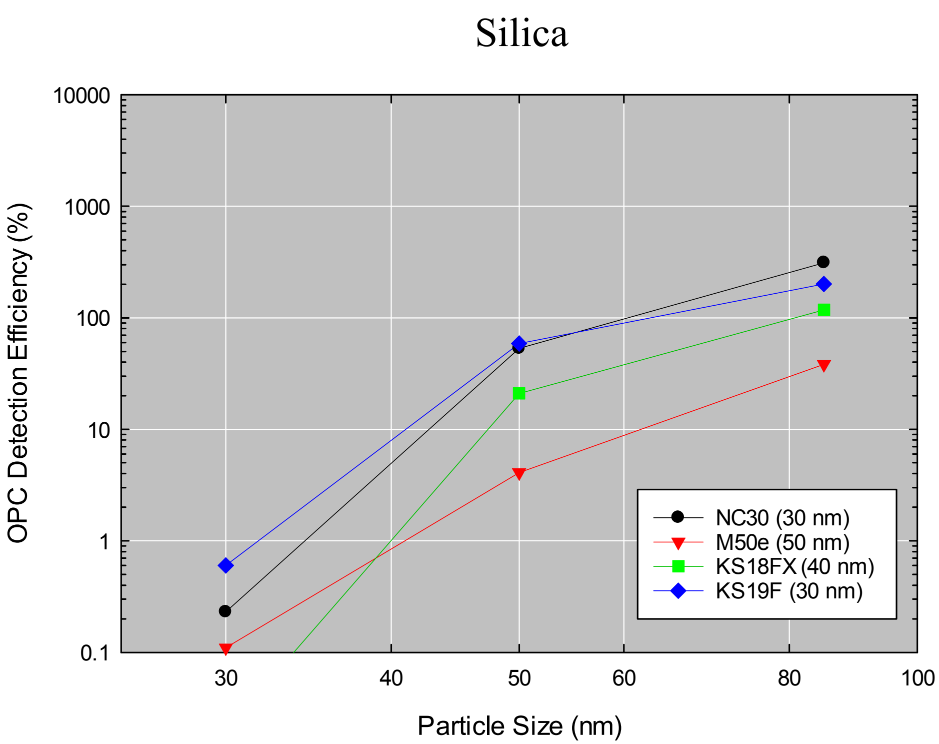


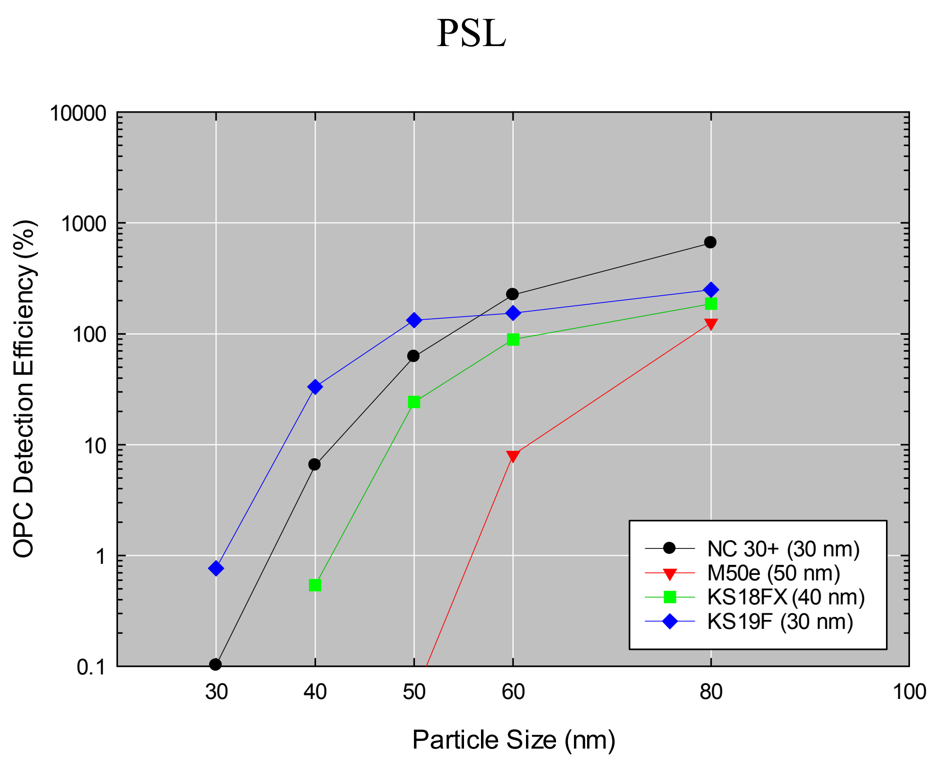
Second Channel Detection Efficiency



- Regular measurement of detection efficiency can be a useful tool for identifying the need for instrument adjustment or calibration.

Silica vs PSL Counting Efficiency

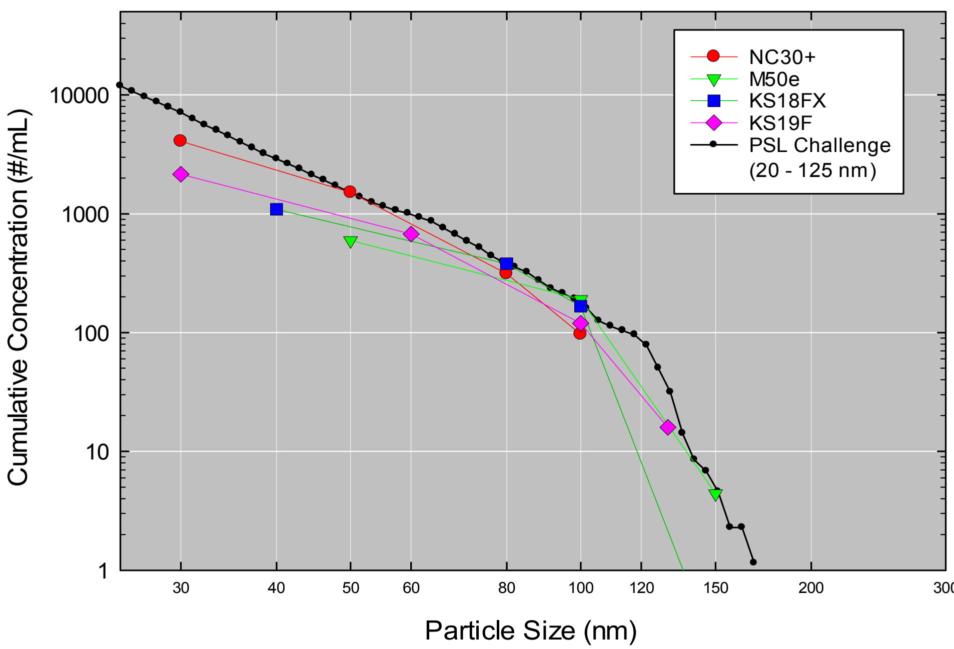




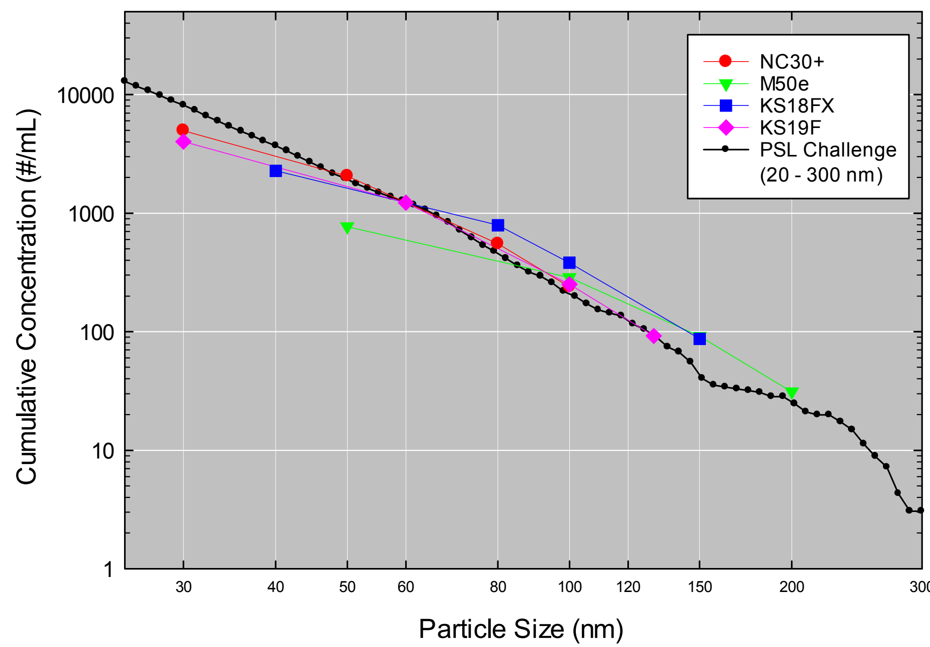
- The OPCs tested have a similar but lower detection efficiency with silica compared to PSL.
- It appears that there may not be an equivalent change in the OPC response to different sizes of silica and PSL, but limited data prevents drawing a conclusion here.

OPC response to polydispersed PSL

20 to 125 PSL Poly



20 to 300 PSL Poly



- Even though the NC30+ had a lower detection efficiency than the Rion KS19F for PSL ≤ 60 nm, it had better correlation to the polydispersed challenge.
- The addition of larger PSL resulted in over-counting by some of the counters.

Observations and Conclusions

- Measuring particles in liquids below 100 nm is quite challenging, even when particles are of know composition and the liquid is ultrapure water.
- Each of the OPCs tested responded differently to monodispersed PSL, polydispersed PSL and silica challenges.
- The fact that they respond differently does not mean that one is right and another wrong. It means that it is important to understand the response of the particular instrument you are using.
- Determining OPC detection efficiency and response to polydispersed PSL is a tool that should be considered when comparing data generated by different instruments.

Observations and Conclusions

- Preparing PSL sizing standards for OPC detection efficiency measurement requires a complete understanding of the PSL particle size distribution, particularly below 100 nm.
- With appropriate PSL standards, OPC detection efficiency can be quantified.
- Polydispersed PSL provides insight to the anticipated response of the instrument to more "normal" particle distributions. This may be useful as a quick check of OPC performance.
- The detection efficiency determined using PSL appears to translate reasonably well to "real world" particles such as silica.

Acknowledgments

- Chris Howe and Arik Litchy (CTA) - PSL standards preparations and OPC testing.
- Adam Herrman and Rich Hoffman (Entegris) - Assistance in the setup and operation of the Rion instrumentation.

Footnote - PSL and silica concentration standards are commercially available from Kanomax FMT.

Thanks for your attention!