Technique for measuring the particle retention of liquid filters to 10 nanometers

Gary Van Schooneveld and Don Grant
CT Associates, Inc.
7121 Shady Oak Road, Eden Prairie, MN 55344
gary@ctassociatesinc.com

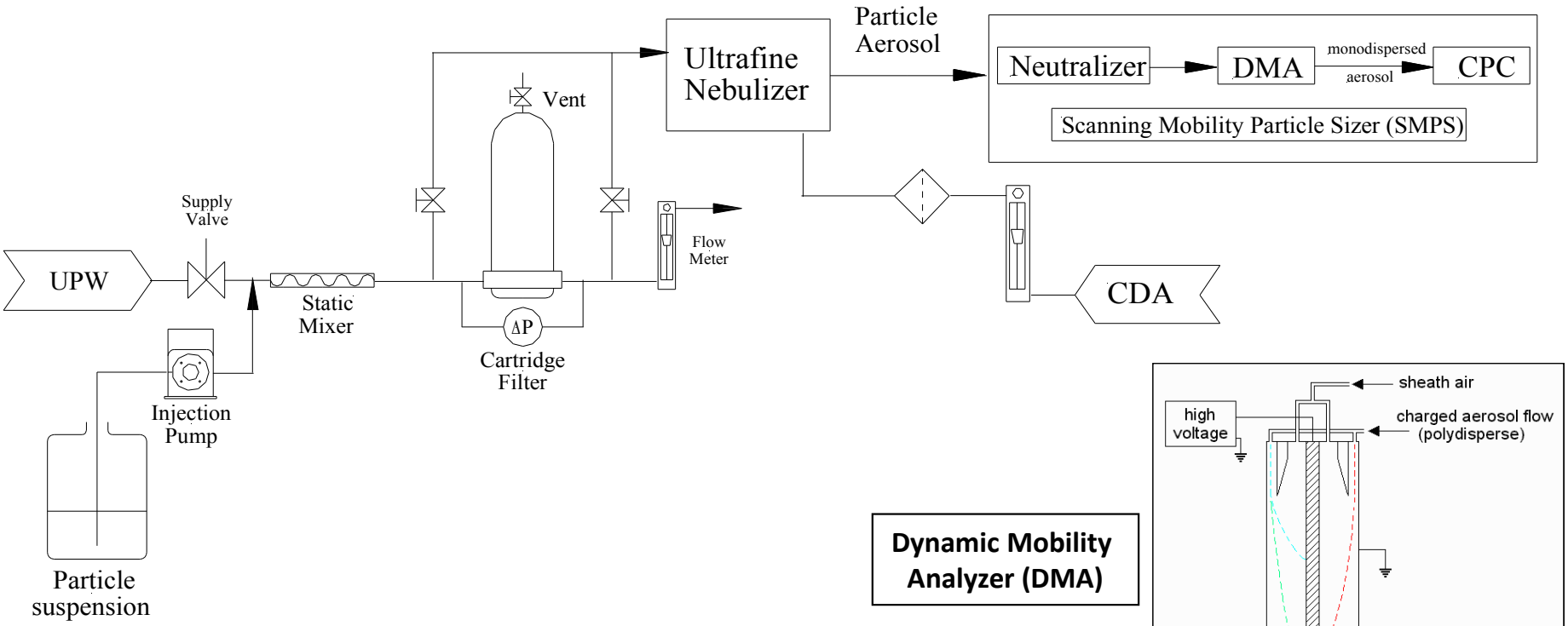
Introduction

- Industrial requirements have driven the particle retention capabilities of liquid microfiltration and ultrafiltration processes to near or below 10 nanometers.
- International Technology Roadmap of Semiconductor (ITRS) has defined a critical particle size of 20 nm for 2013 and projects that in 2019, the critical particle size will be 10 nm.
- Optical particle counters traditionally used for measuring retention of ultrahigh-purity water (UPW) and chemical filters are beginning to reach their lower limits and are currently not able to measure below 30-40 nanometers.
- The potential interaction between the test particles such as polystyrene latex (PSL), gold and silica used for the challenge and the filter media is not well established.
- This presentation will discuss the use of a liquid nanoparticle sizing system capable of measuring both the size and concentration of nanoparticles in ultrapure water and its application for testing high-purity liquid filters.

Outline

- Review the testing and particle measurement apparatus
- Comparison of candidates particles used for retention testing; in particular, their particle size distributions (PSD).
- Retention of candidate test particles by a 30 nm UPW filter
- Examples of filter retention using mono- and poly-distributed silica
- Summary

Liquid nanoparticle sizing apparatus - filtration

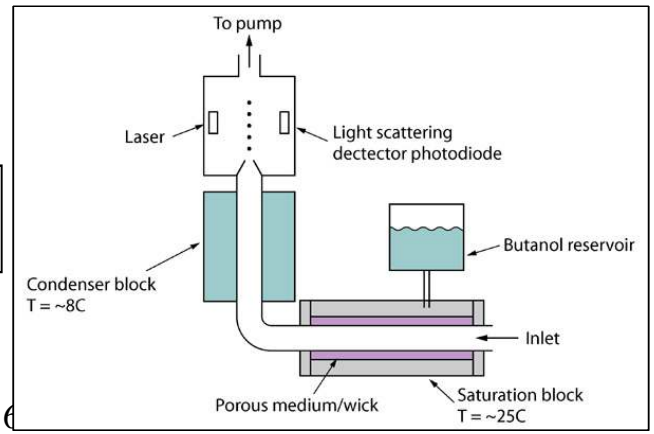


Dynamic Mobility Analyzer (DMA)

Operating Principle

- Nebulizer converts the hydrosol to an aerosol.
- DMA separates particles according to size.
- CPC measures concentrations of particles of each size.

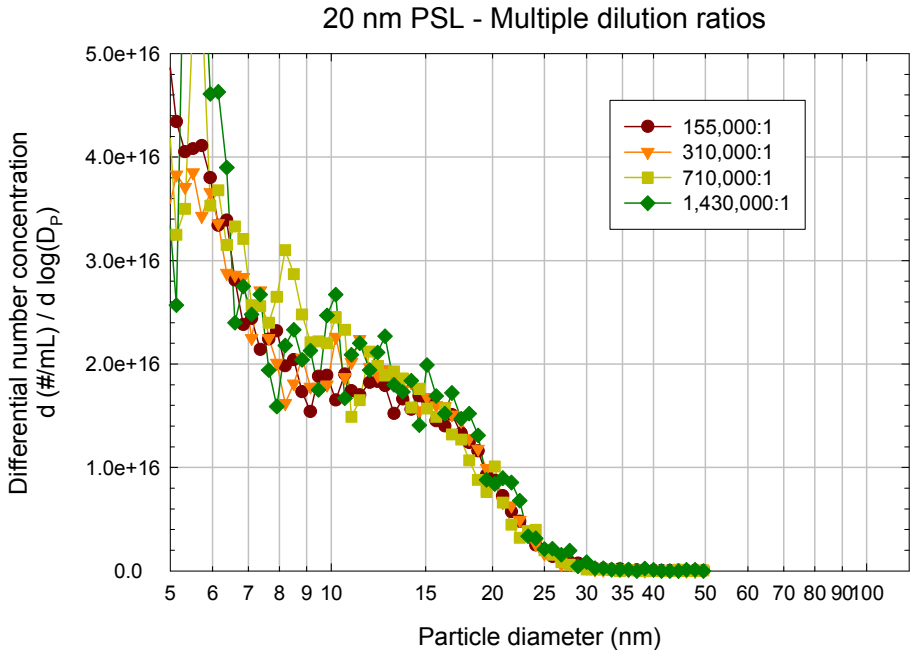
Condensation Particle Counter (CPC)

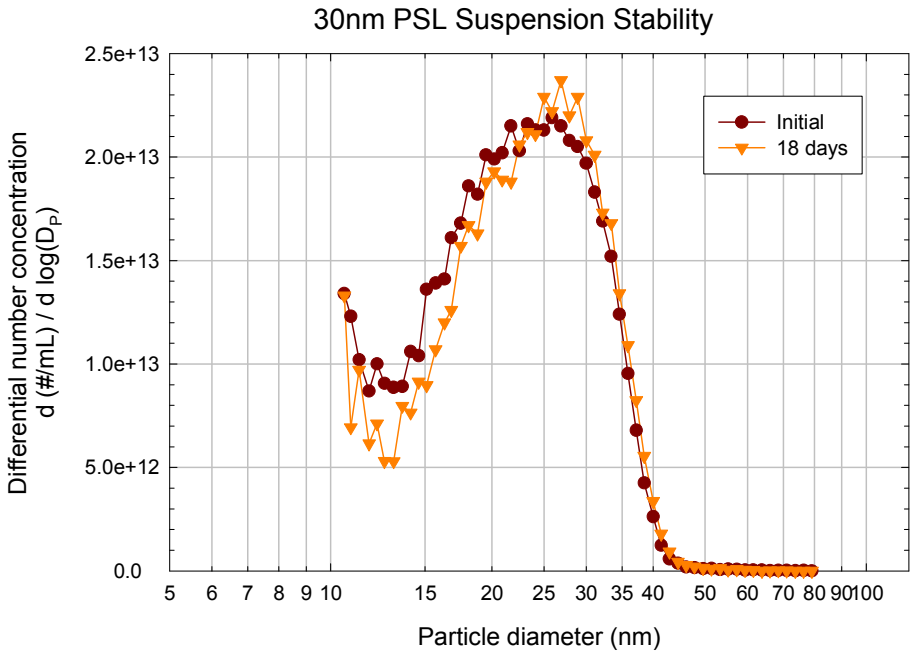


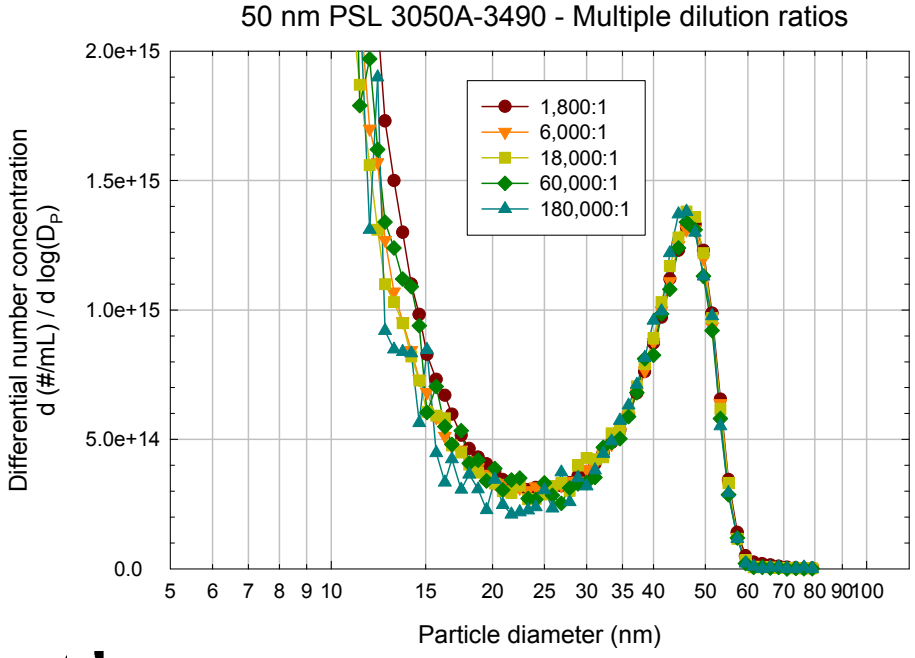
Liquid nanoparticle sizing system capabilities and benefits

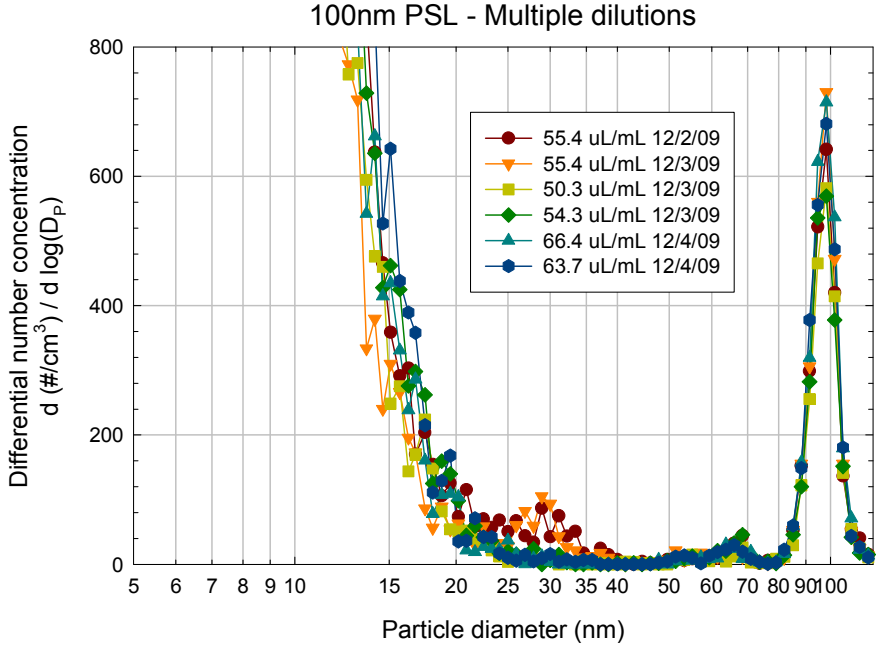
- Broad operational range:
  - Single-particle quantitation from samples with particle concentrations at 2×10^8 - 1.0×10^{15} particles/mL .
  - Sizing range from 2.5 to 450 nm with 64 channels per decade resolution (scan mode).
  - Cumulative concentration detection limit ≥ 10 nm of 2×10^6 particles/mL (fixed channel mode).
- Actual concentrations, not relative concentrations are measured.
- Shape of the particle size distribution is not assumed- able to resolve multi-modal distributions.
- Highly sensitive to small changes in the PSD.
- Technique is independent of the optical and density properties of the particles.
- Suitable for on-line or off-line measurement.

Examples of PSL particle size distributions[1]

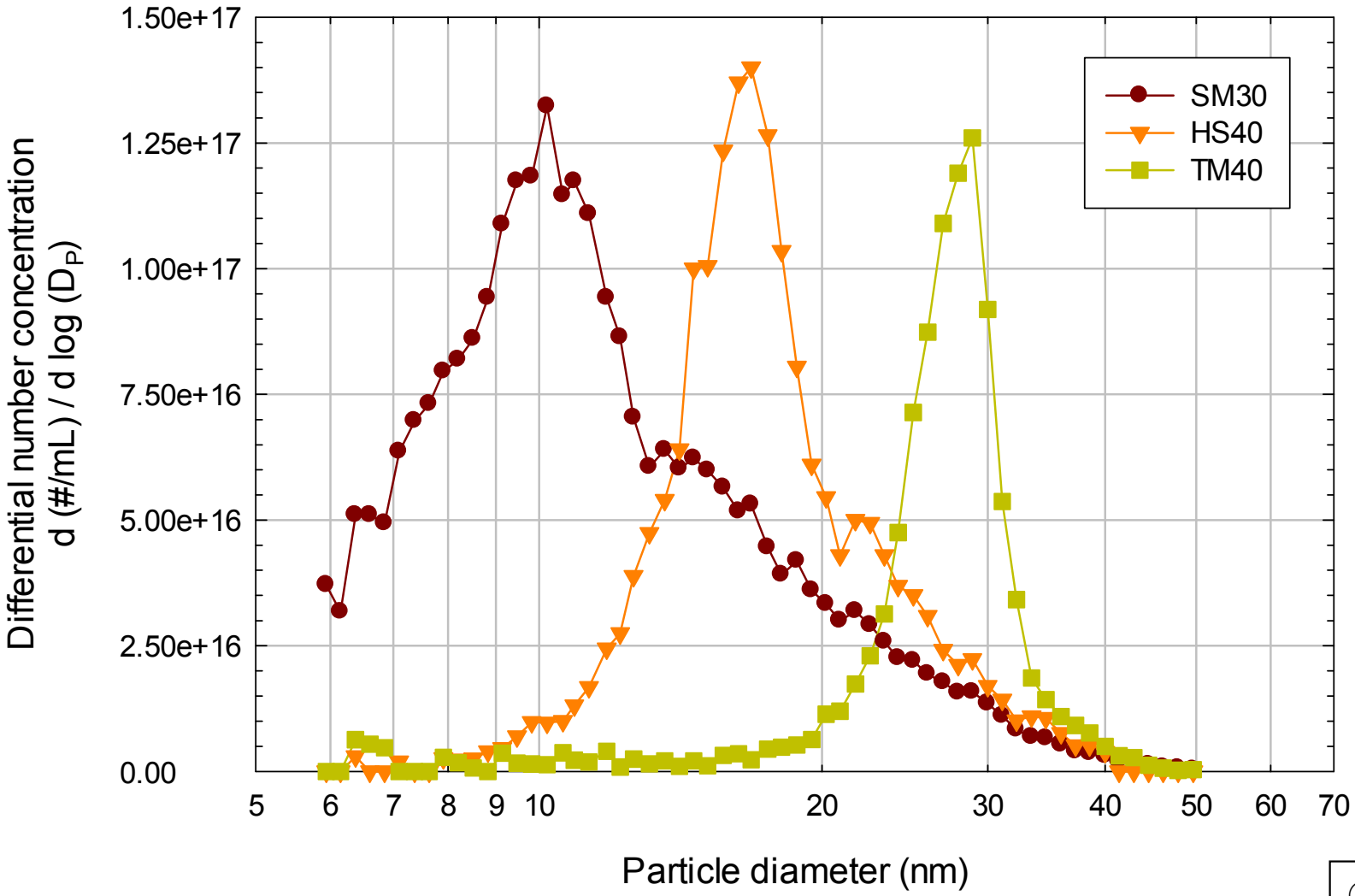






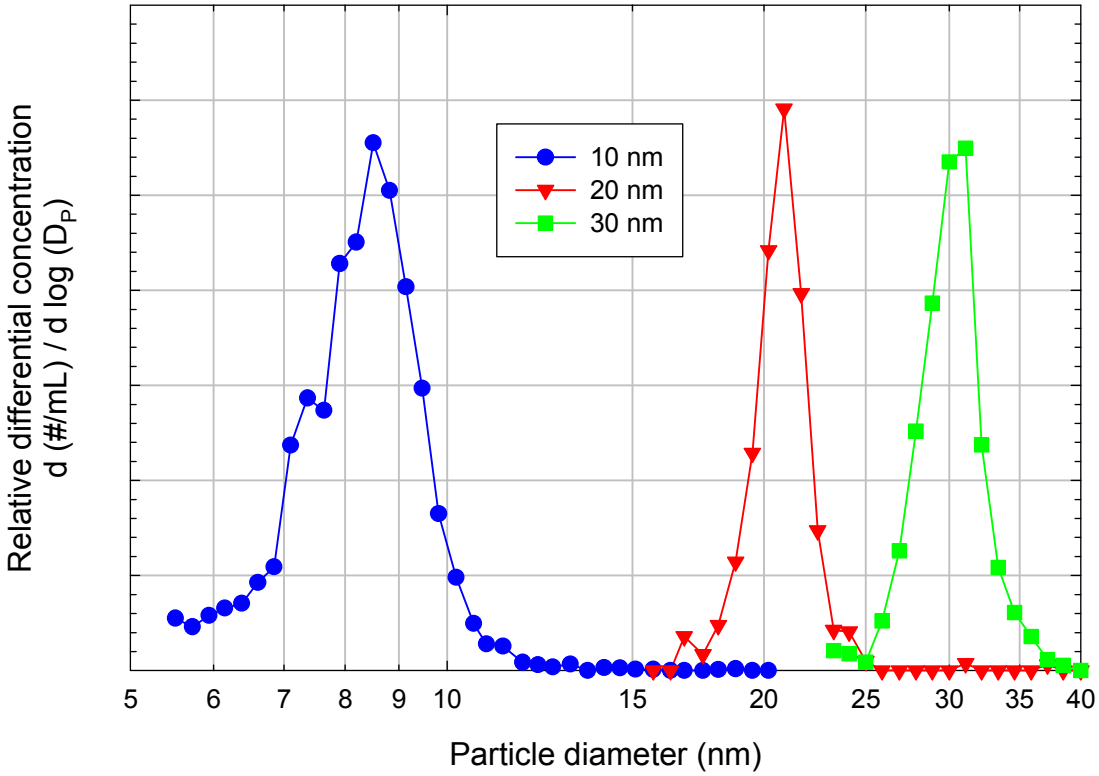


Size distributions of Ludox® Colloidal Silica Particles[1]



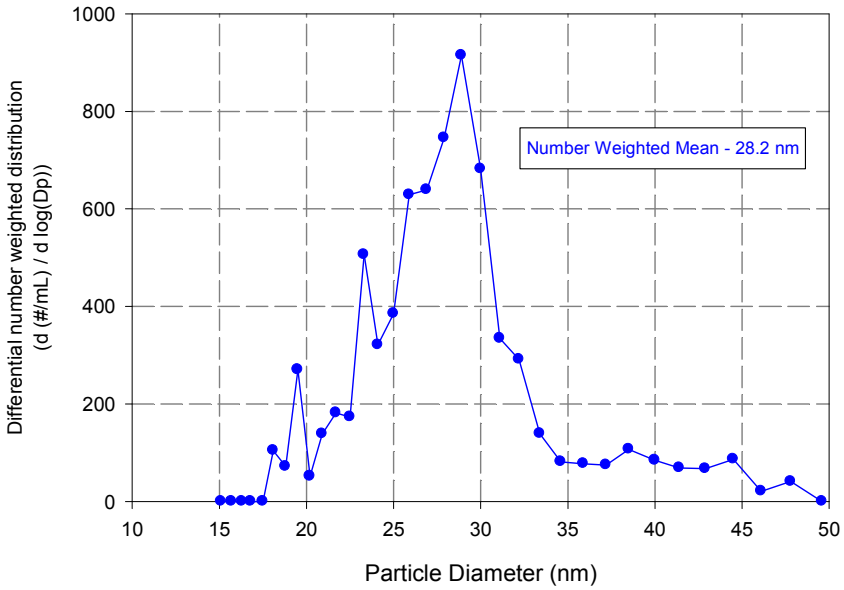
® Grace Davison

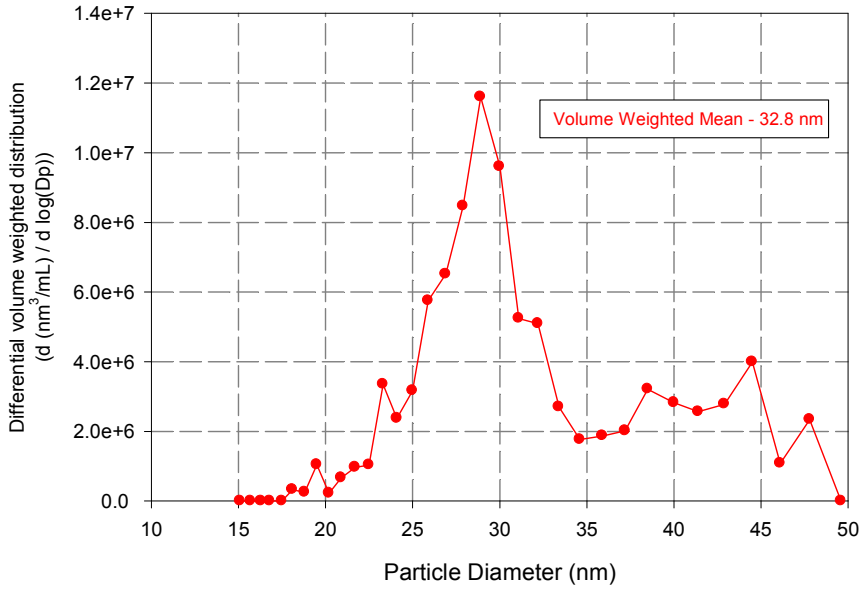
Size distributions of gold nanoparticles[1]



Nominal Size (nm)	Claimed size (TEM)		Measured size (LNS)	
	Mean (nm)	CV (%)	Mean (nm)	CV (%)
10	9.3	< 15	8.4	13
20	20.3	< 8	20.8	7.4
30	30.3	< 8	30.5	7.3

NIST 30 nm gold analysis via liquid nanoparticle sizing





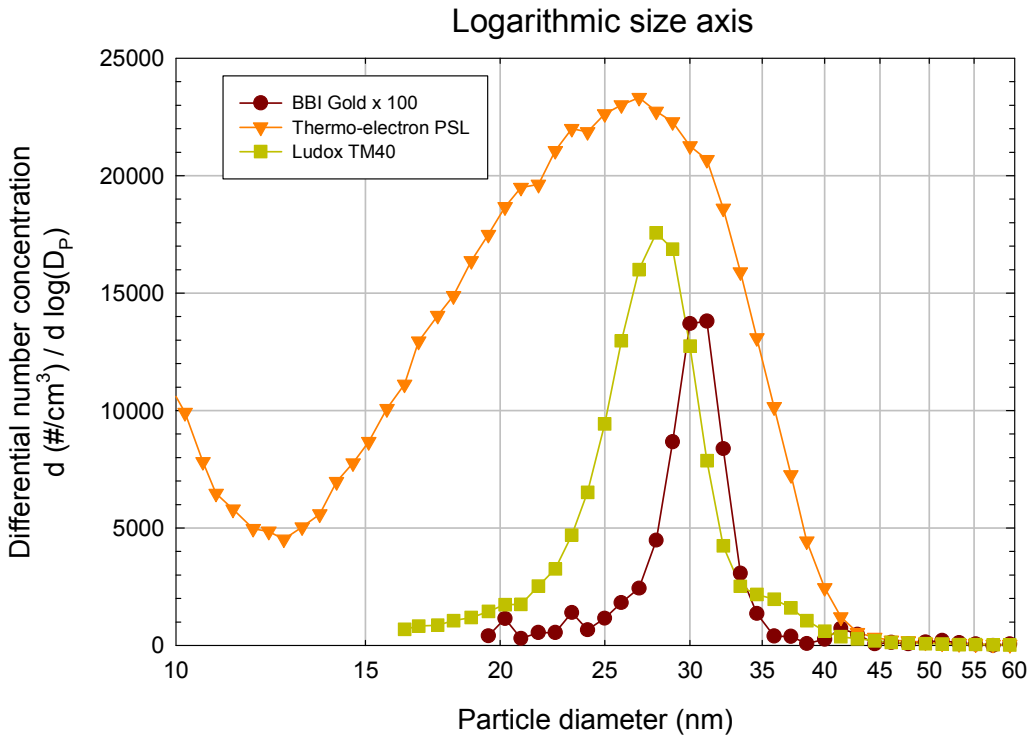
NIST Report of Investigation – Reference Material 8012 – 30 nm Gold Nanoparticles

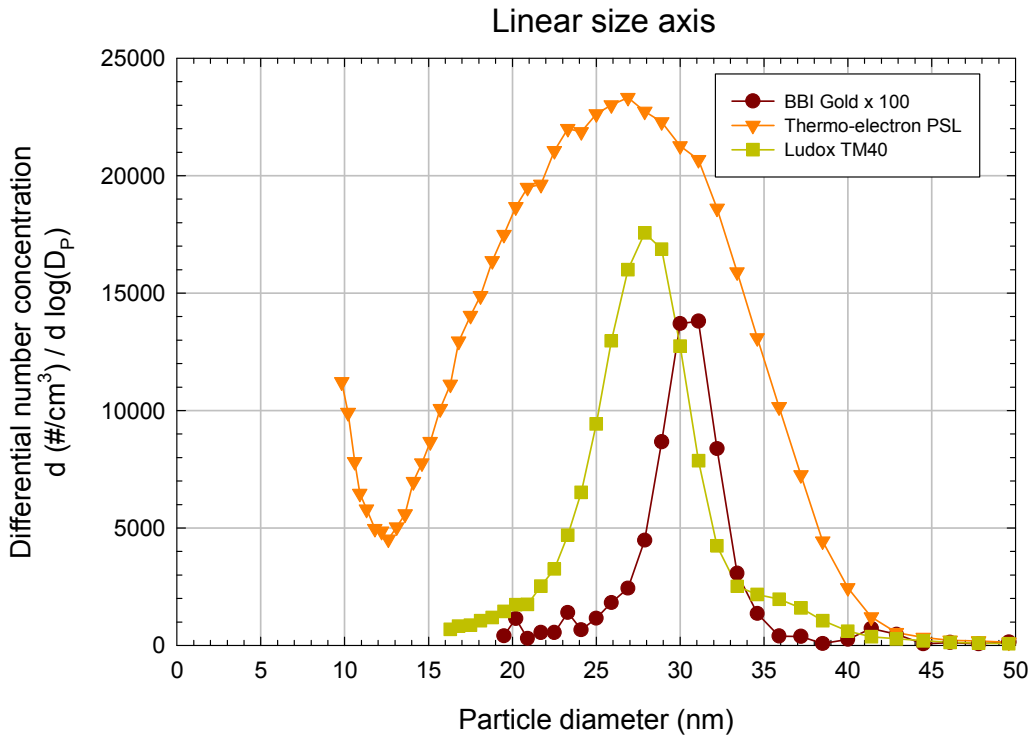
Technique	Analyte Form	Mean Particle Size (nm)		Expanded Uncertaincy
Atomic Force Microscopy	dry, deposited on substrate	24.9	±	1.1
Scanning Electron microscopy	dry, deposited on substrate	26.9	±	0.1
Transmission Electron Microscopy	dry, deposited on substrate	27.6	±	2.1
Differential Mobility Analysis	dry, aerosol	28.4	±	1.1
Dynamic Light Scattering	liquid suspension			
173° scattering angle (back scattering)		28.6	±	0.9
90° scattering angle		26.5	±	3.6
Small-Angle X-Ray Scattering	liquid suspension	24.9	±	1.2

Study #1: 30 nm filter retention testing using three types of 30 nm particles

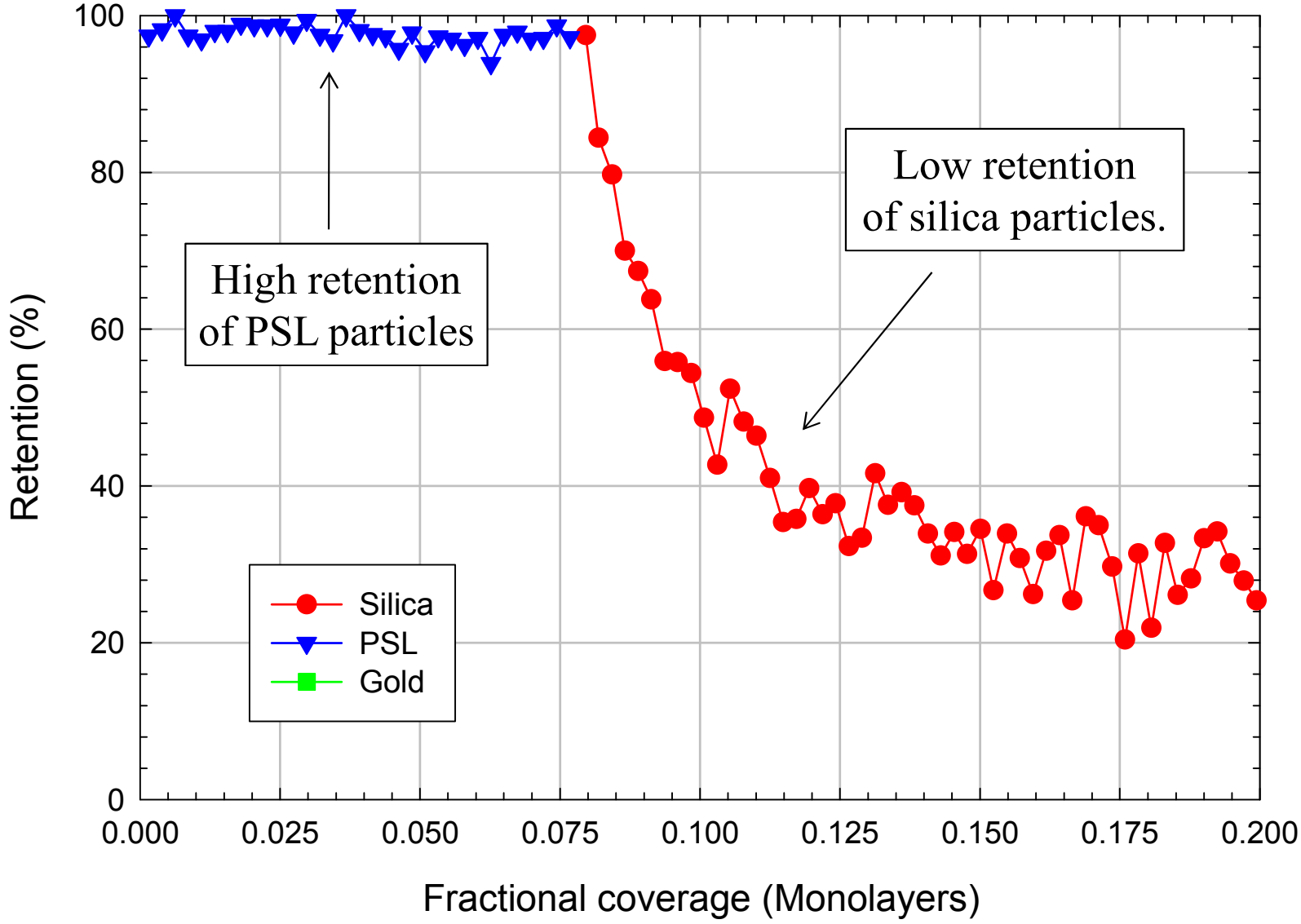
- Three, commercial 30 nm rated 10-inch cartridge filters from one manufacturer were tested.
- The cartridges were flushed until the filtrate approached the system background concentration (~10^6/mL > 10 nm).
- The filter was challenged with 2E8 particles/mL (~6 ppb).
- Testing conducted using single channel mode (cumulative concentration ≥ 10 nm)
- The UPW face velocity throughout the test was 0.11 cm/min.

PSDs of 30-nm particles



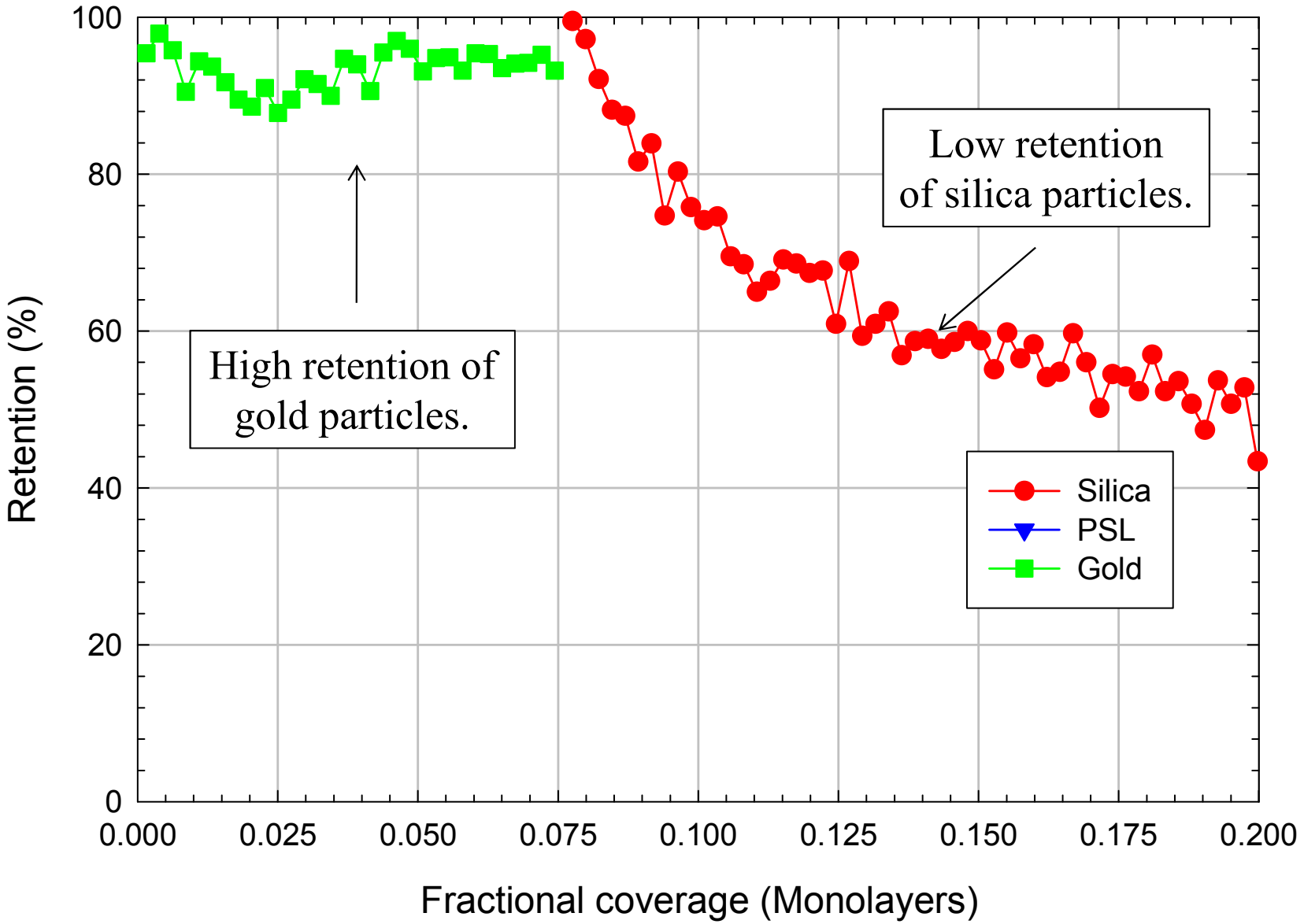


Retention of PSL followed by silica
(Cartridge #1)

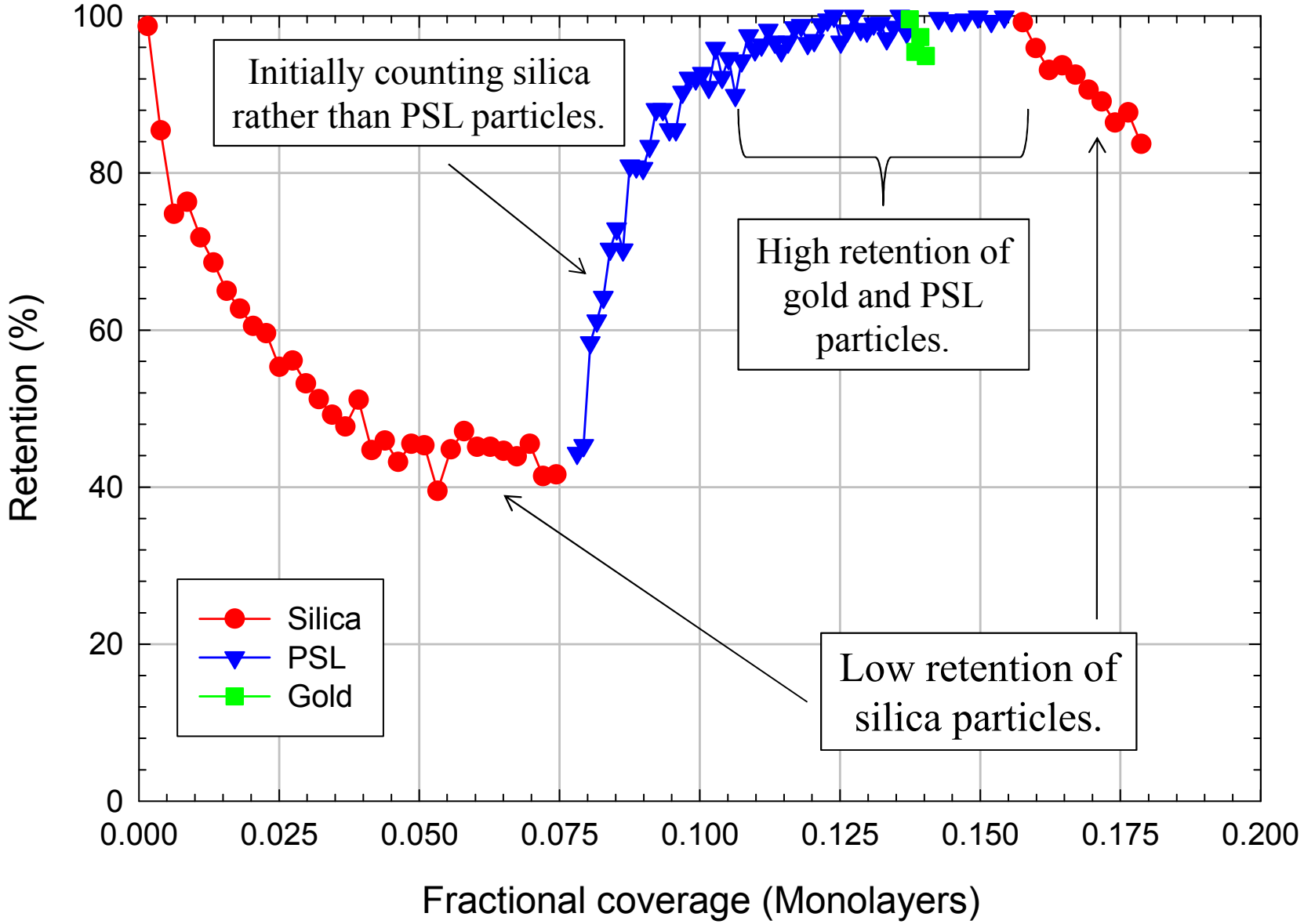


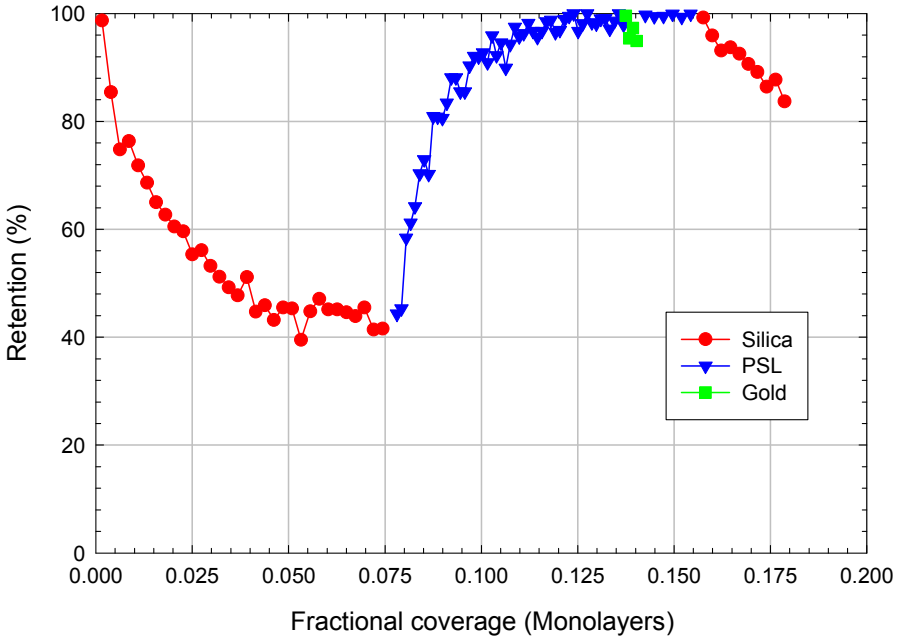
Retention of gold followed by silica

(Cartridge #2)



Retention of multiple particle types
(Cartridge #3)



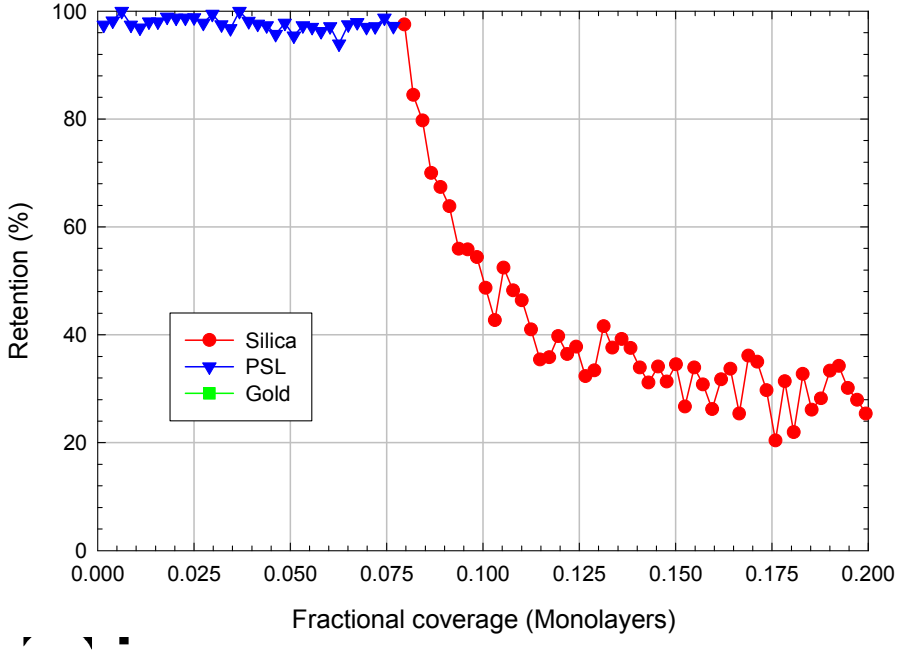


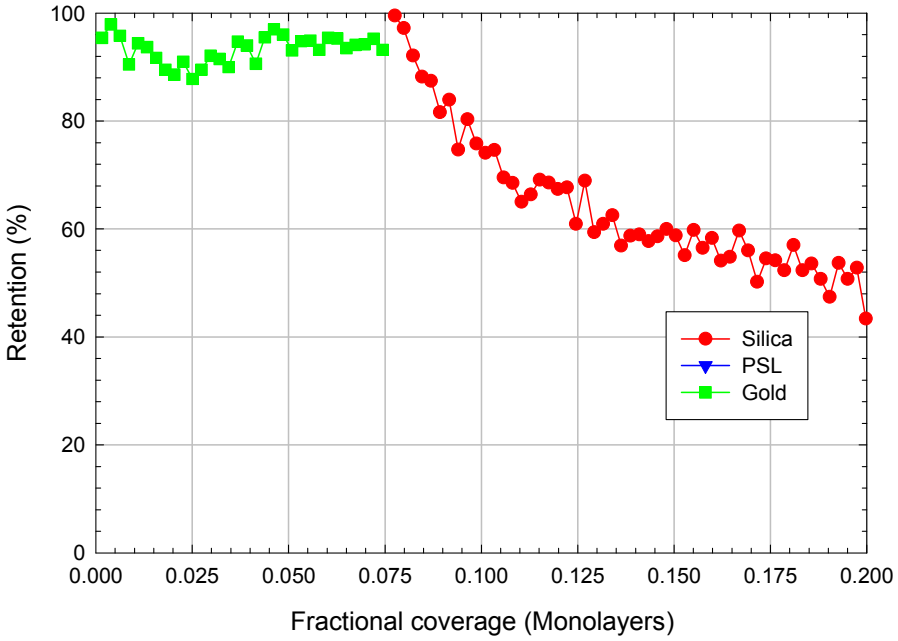
Observations

- Capture mechanism(s) other than sieving are active with gold and PSL that are not with silica. It is desirable to limit the capture mechanism to sieving.
- Silica is a particle that is found in water systems while gold and PSL are not.
- Silica is much less expensive than gold or PSL.

Recommendation

- Use silica for retention testing.

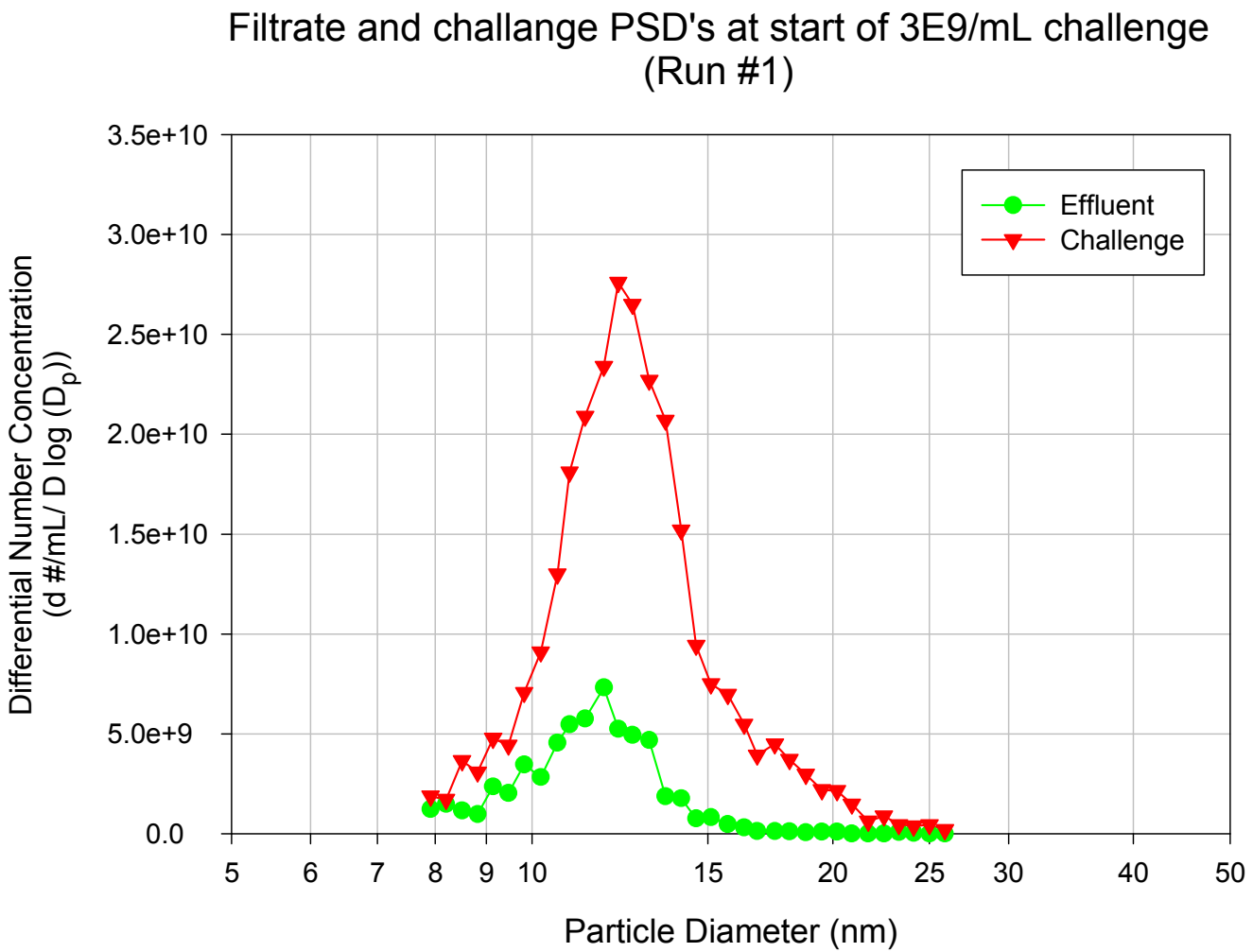




Study #2: Filter capture of mono-distributed colloidal silica particles

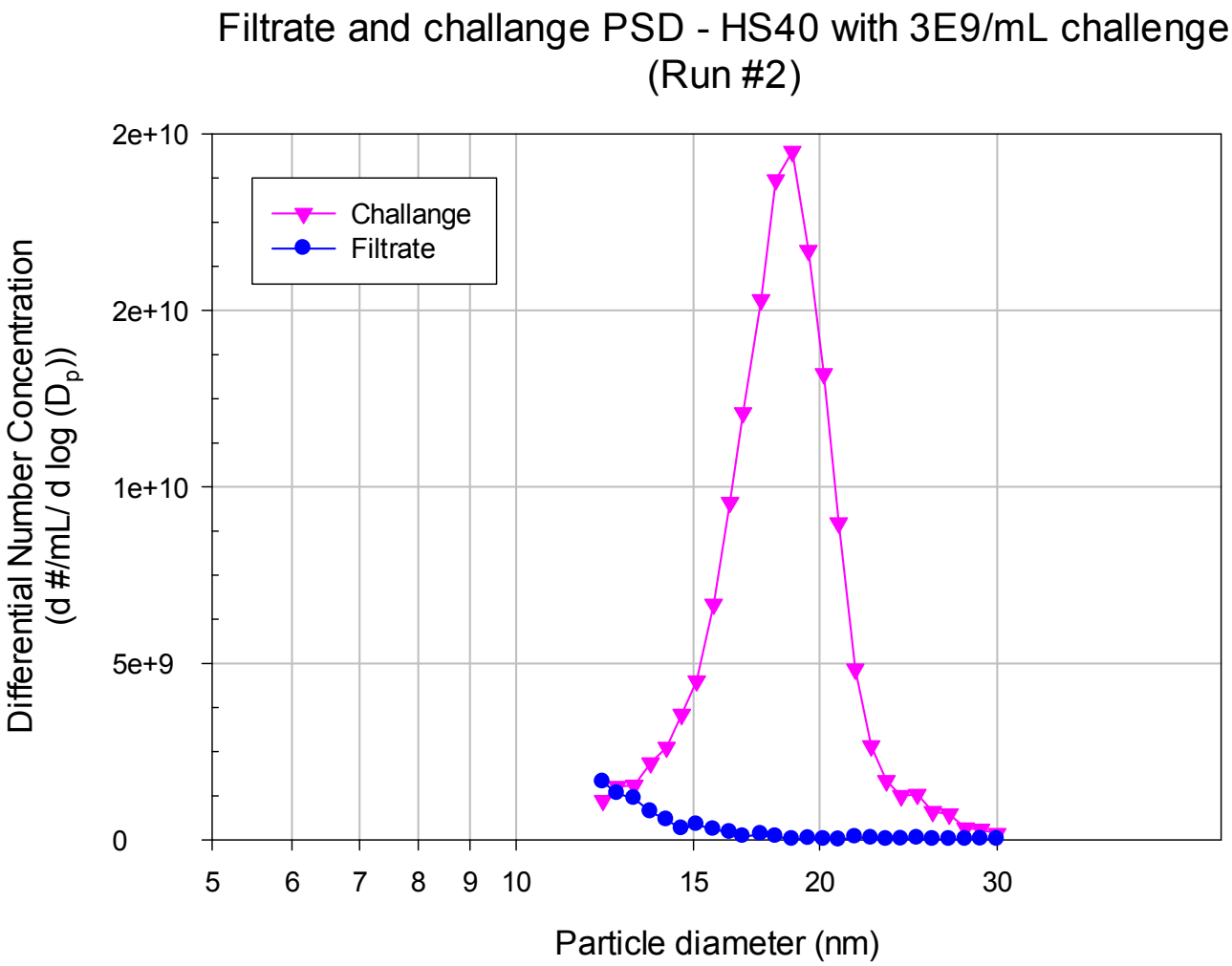
- Two filters tested; same pore size rating and same manufacturer.
- One filter challenged with 12.6 nm mono-distributed silica particles, the other with 18.8 nm mono-distributed silica.
- Prior to the particle challenge, the filters were flushed with UPW to reduce particle shedding.
- $3x10^9$/mL particle concentration was used.
- Particle concentrations measured upstream and downstream of the filter (scan mode).
- Filter particle retention was calculated as a function of particle size.

Filter retention as a function of particle diameter.



Test method work conducted in support of developing SEMI test guidelines for measuring retention of 5-15 nm filters used with UPW.

Filter retention as a function of particle diameter.



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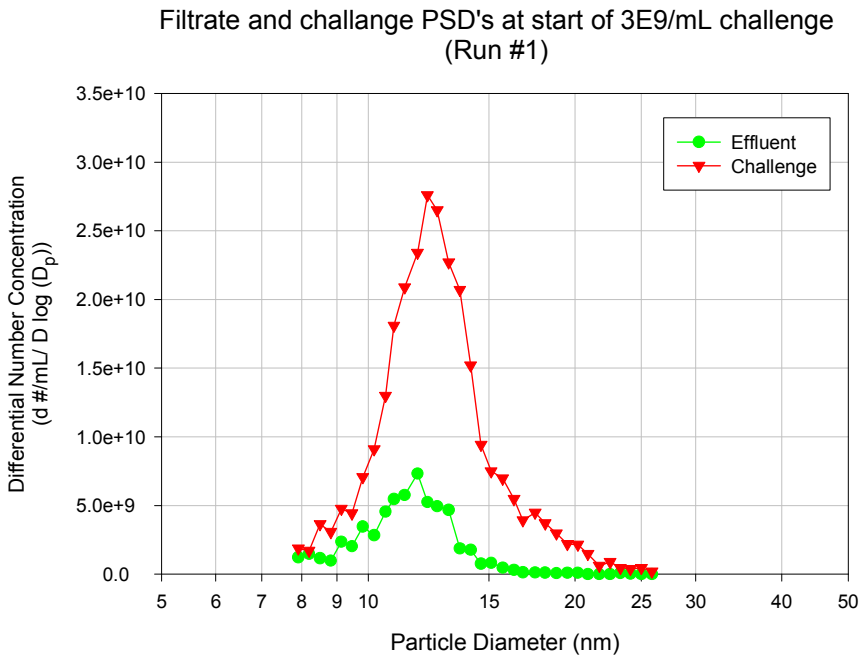
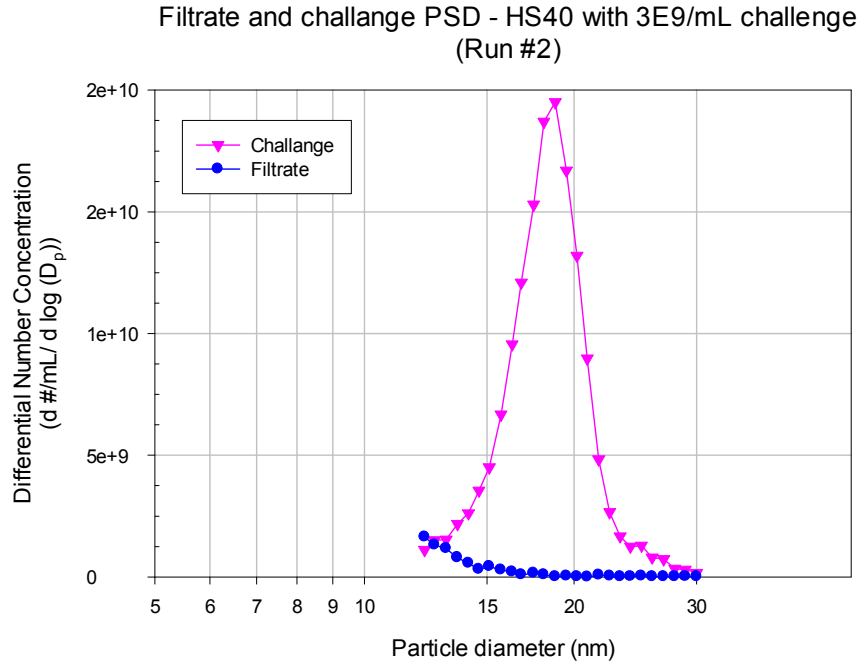
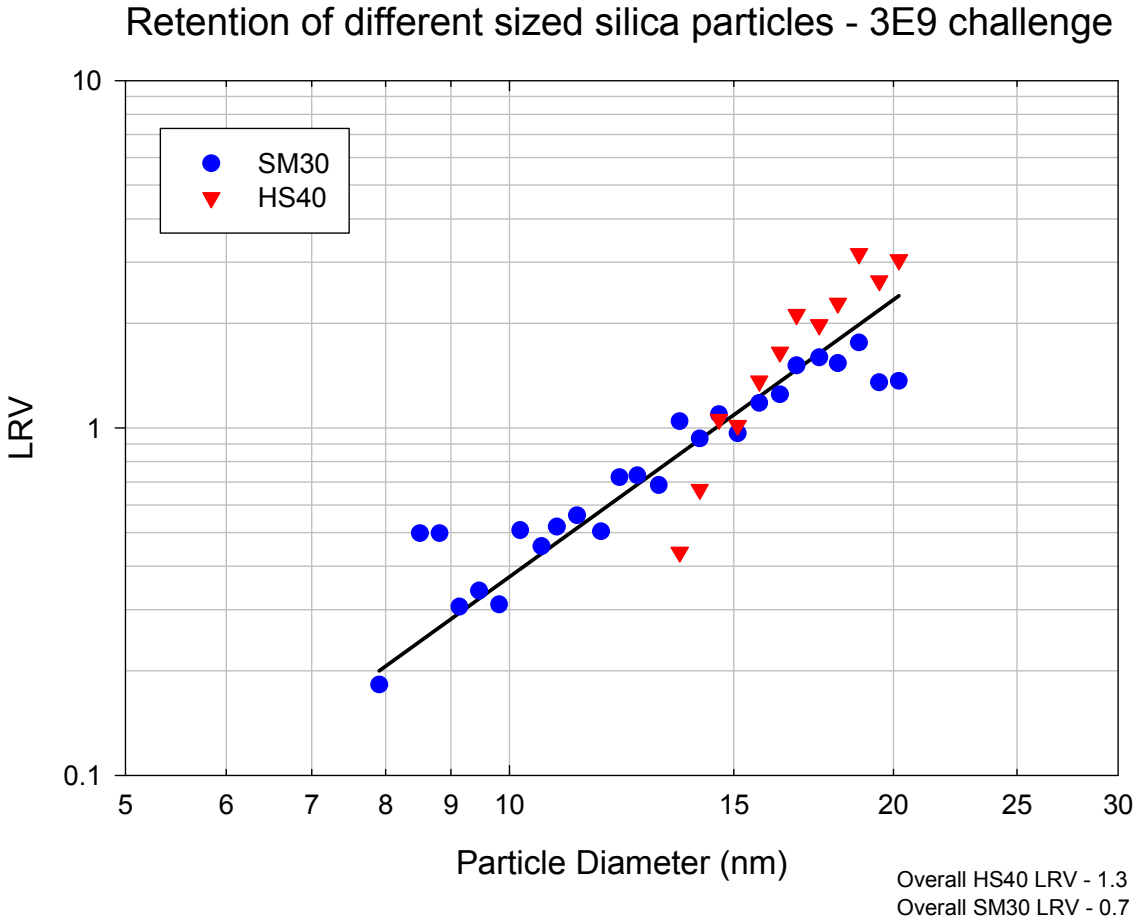
Filter retention as a function of particle diameter.





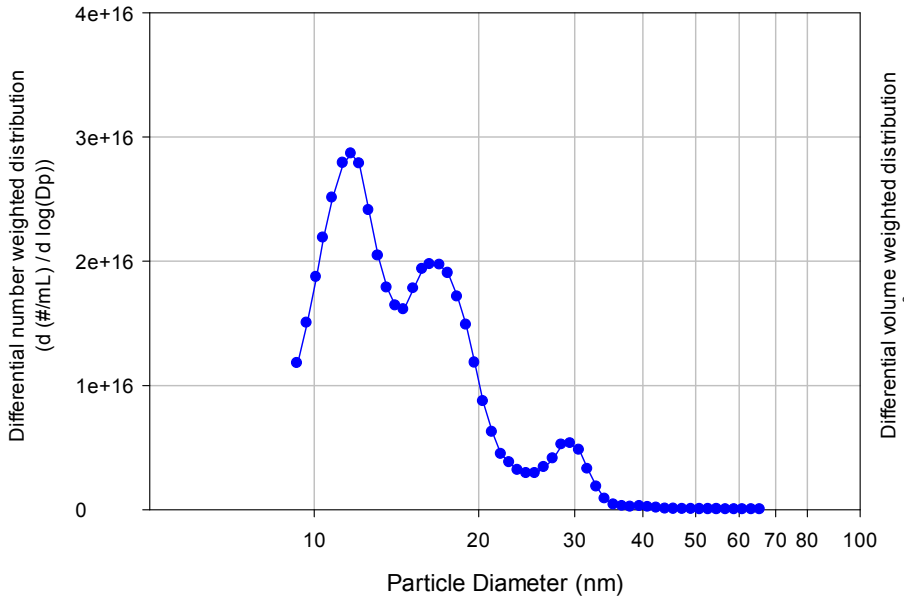


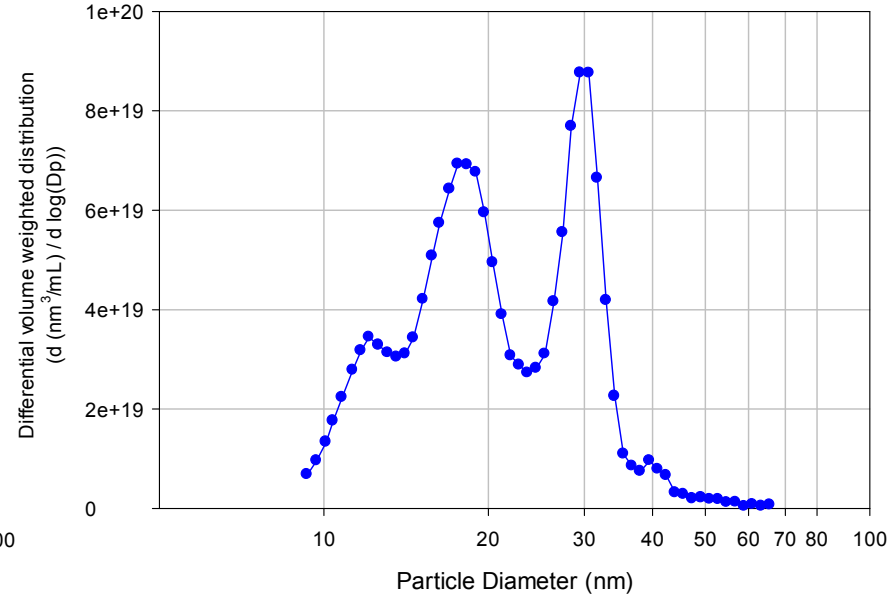
Test method work conducted in support of developing SEMI test guidelines for measuring retention of 5-15 nm filters used with UPW.

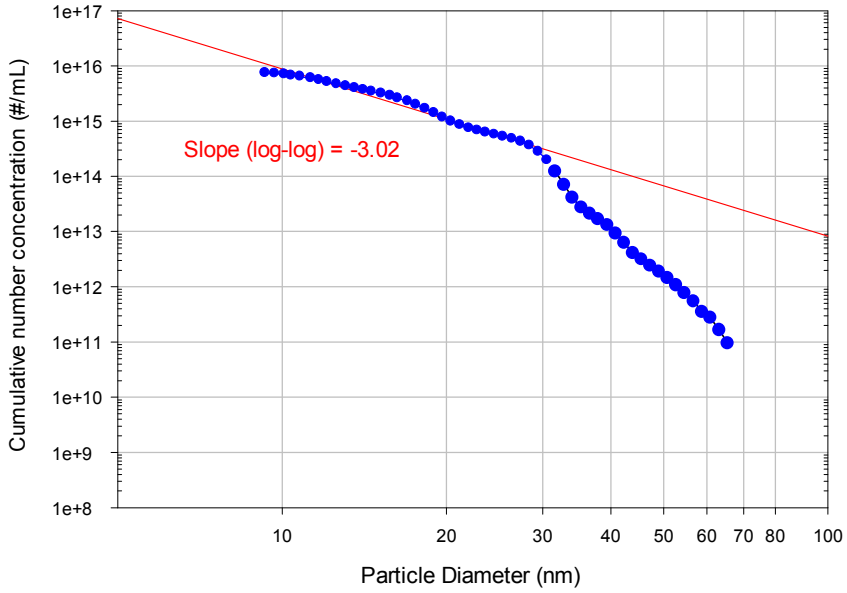
Study 3: Filter capture of poly-distributed colloidal silica particles

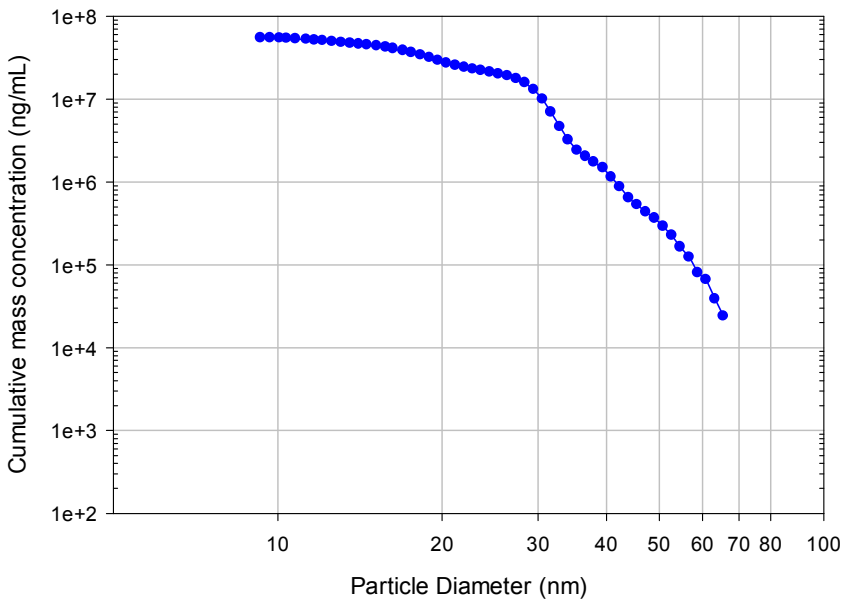
- Filter retention test using poly-distributed silica in UPW.
- Three silica size mixture: 12, 18 and 28 nm.
- Flush filter with UPW to reduce particle shedding. Measure particle concentration during rinse.
- $5x10^9$/mL ≥ 10 nm particle concentration was used.
- Measure particle concentrations upstream and downstream of the filter (scanning mode).
- Filter particle retention was calculated as a function of particle size and loading.

Silica challenge particle size distribution



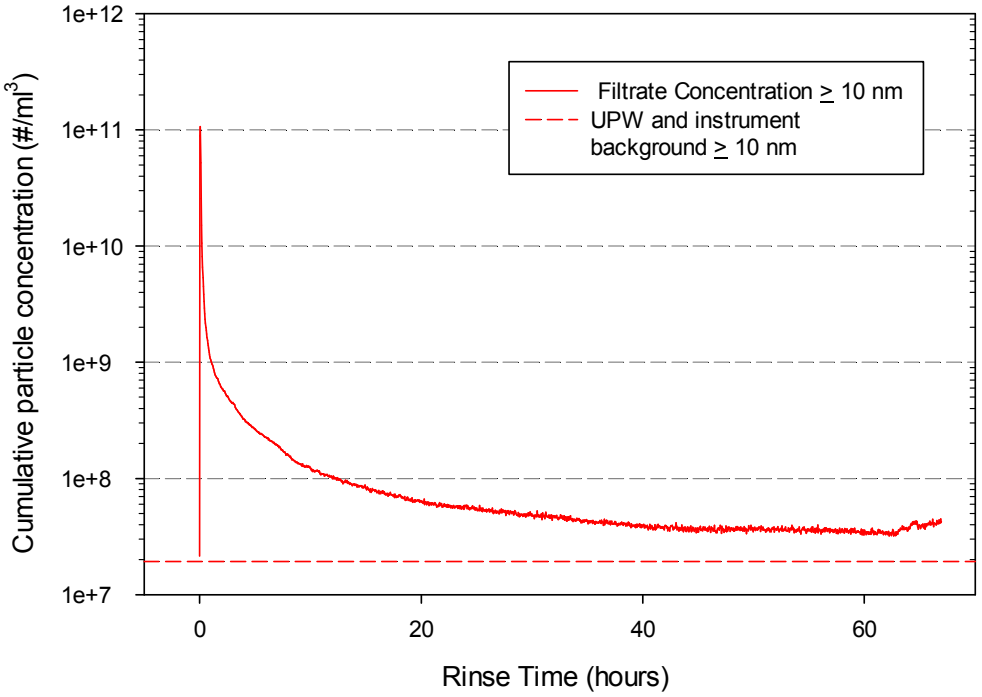




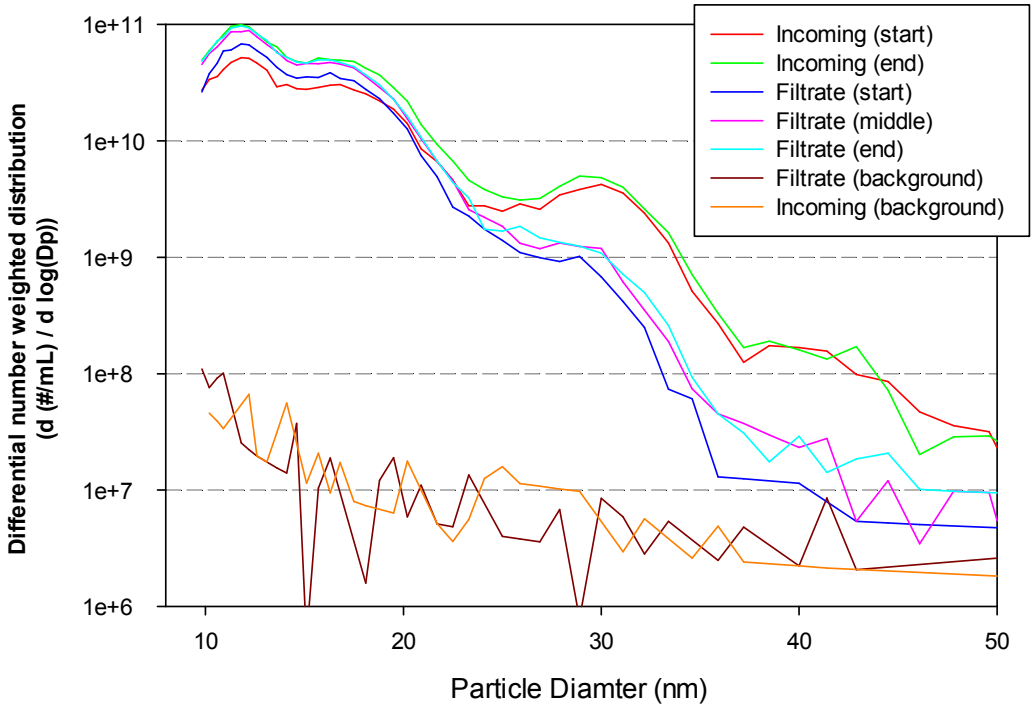


Filter Test Results

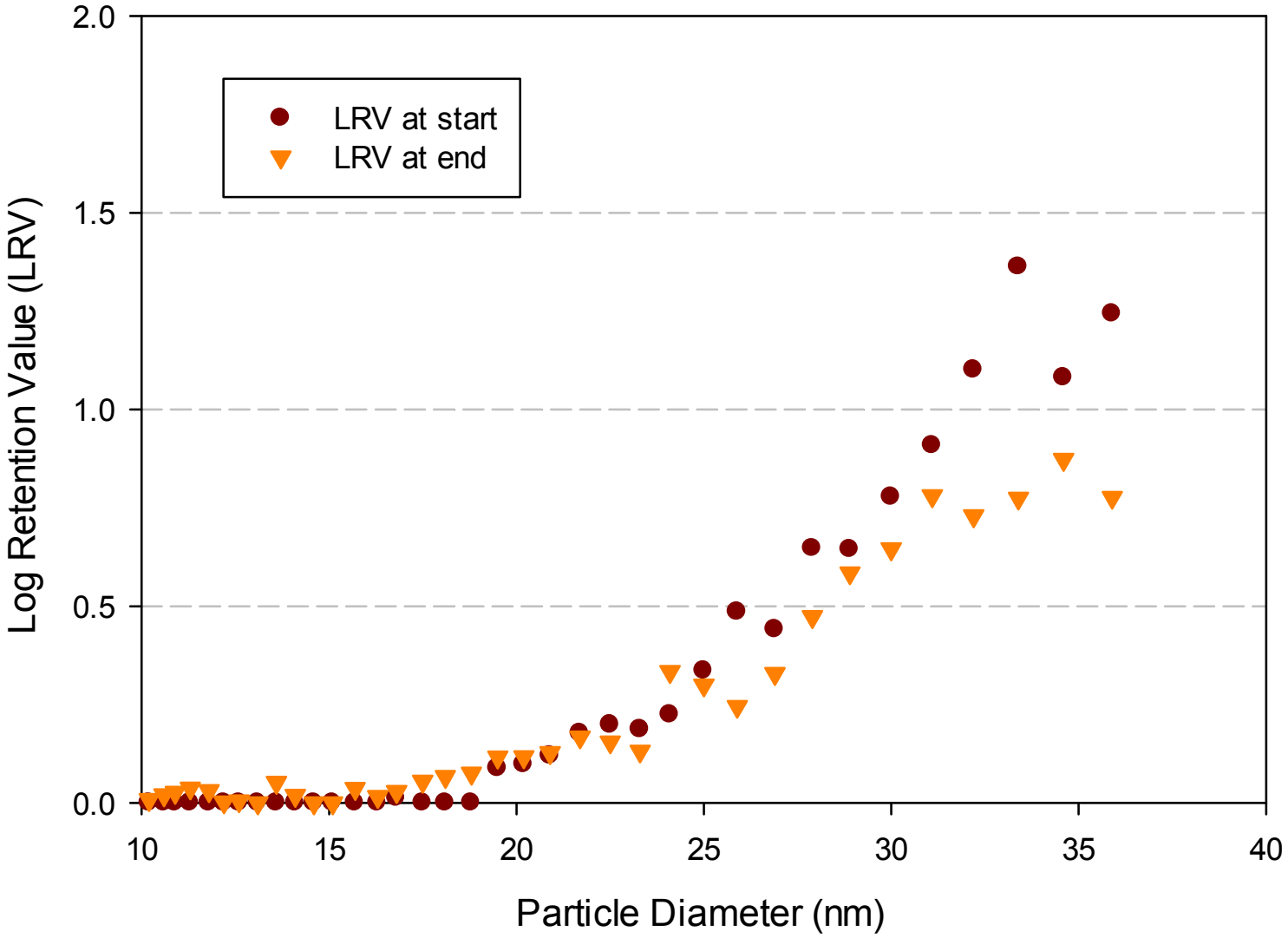
Filter rinse in UPW



Retention Data



Filter retention analysis



Summary

- Instrumentation and filter test methods have been demonstrated for the measurement of particle retention in liquid filters to 10 nanometers.
- Various nanoparticles were investigated and assessed for suitability as filter retention challenge materials.
- In the case of UPW filter retention testing, silica nanoparticles are recommended.
- Retention relative to particle diameter can currently be measured down to 10 nanometers.

Acknowledgements

- Don Grant – CT Associates
- Erik Willis and TSI

References

(1) Grant DC (2011). "The effect of particle composition on filter removal of sub-30nm particles from UPW," presented at the Executive Forum at ULTRAPURE WATER Micro 2011, Portland, OR.