Measuring sub-50nm particle retention of UPW filters

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Introduction

- The critical feature size of state-of-the-art semiconductor devices is on the order of 40 nm and expected to decrease to < 20 nm by 2015.
- Particles half the size of critical features can reduce finished device yield and reliability.
- Particles in UPW that contacts wafer surfaces during processing can deposit on the wafer surface.
- Microfilters and ultrafilters are used to remove particles from these liquids.
- Test methods are needed to measure the filter particle removal efficiency of particles smaller than 50 nm.

Outline

- Introduction
- Desired test method characteristics
- Comparison of candidate challenge particle properties
- Examples of retention of candidate test particles by a 30 nm UPW filter
- Summary and conclusions

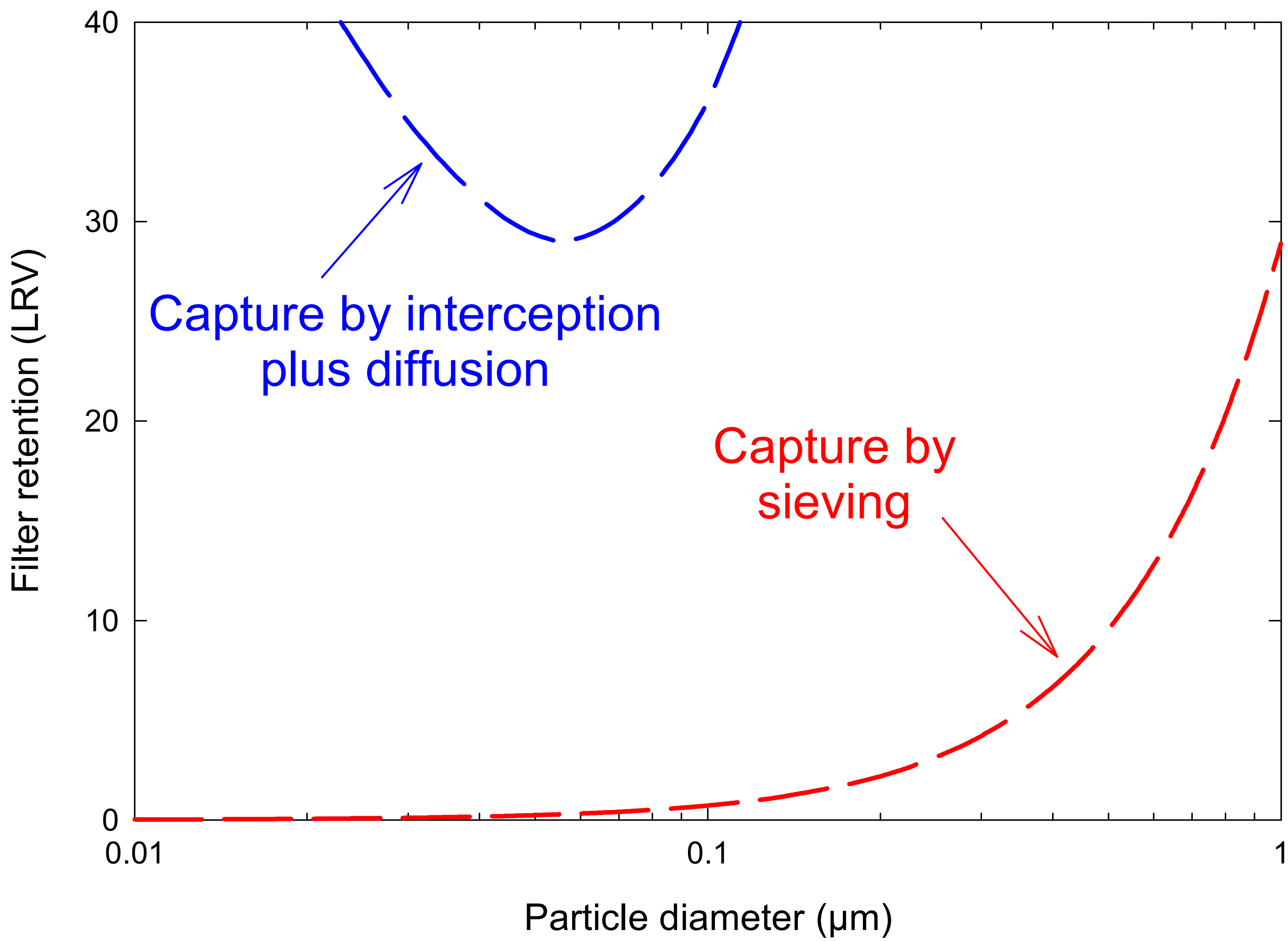
Desired test method properties

- Testing should simulate "real-world" worst case conditions.
  - Particle size should be well characterized.
  - Particle capture should be by sieving only.
  - Particles used in testing should be representative of particles found in UPW systems.
- The test procedure should simulate filter performance during its projected lifetime in a reasonable test period
  - Operate at a face velocity (flow rate/surface area) similar to actual use conditions.
  - Example of a reasonable test duration - simulate 1 year in a 16 hour test.
- The cost per <u>cartridge</u> test should not be prohibitively expensive.

Particle capture mechanisms

- Particle capture by filters can result from several mechanisms including:
  - Diffusion
  - Interception
  - Impaction
  - Electrostatic attraction
  - Sieving
- Particle capture should be by sieving only
  - Worst case capture mechanism
  - Capture by diffusion, interception, impaction and electostatic attraction and adsorption should be absent (or nearly absent).
  - Desire a strong repulsive force and a weak attractive (Van der Waals) force between the particles and the membrane surface to minimize the potential for adsorption.

Removal of particle from UPW by a 0.2 µm rated filter by different capture mechanisms



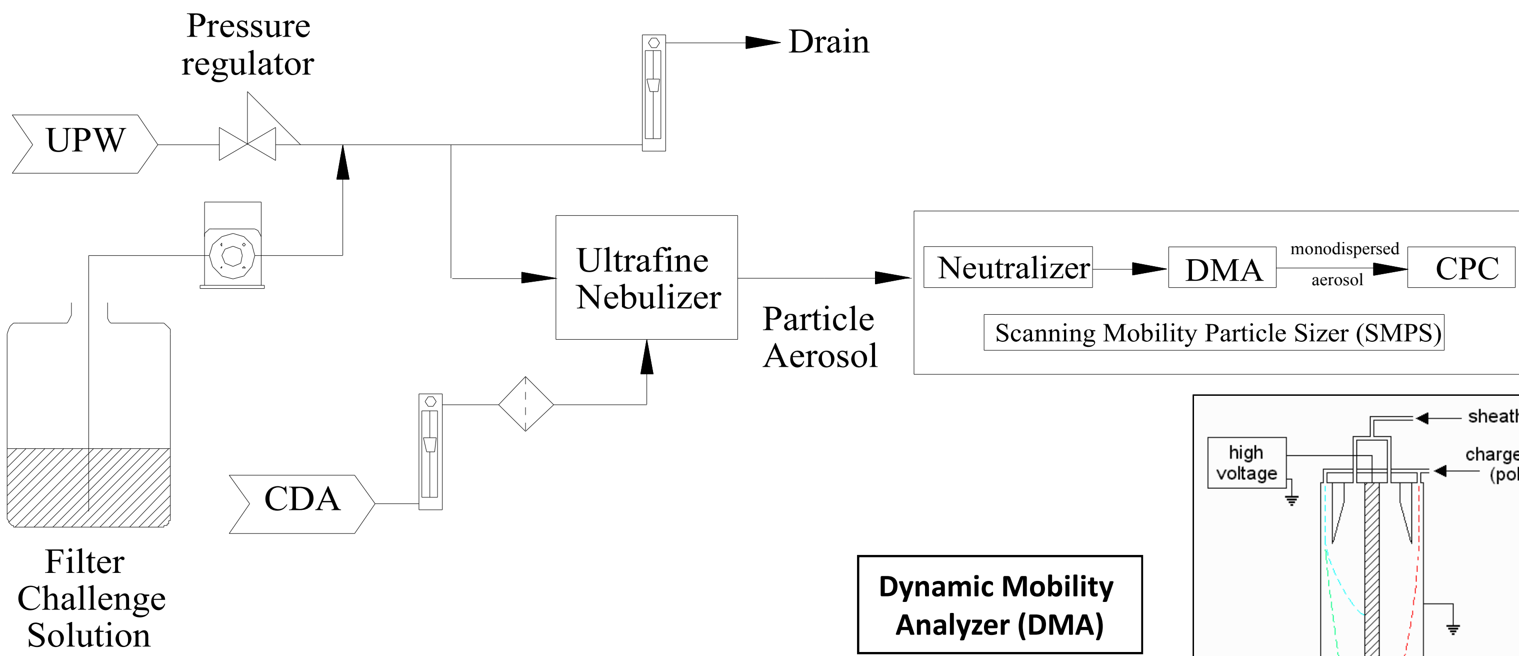
This study

- Focused on particle type.
- Measured the retention of different types of particles by a 30 nm commercially-available UPW filter cartridges.
- Particle types evaluated
  - Polystyrene latex (PSL)
  - Colloidal gold
  - Colloidal silica
- Test conditions employed
  - Filters were operated at a face velocity of 0.11 cm/min (equivalent to ~1.1 liters/min in a 10” cartridge).
    - Lower than actual use conditions.
    - Chosen due to the high cost of gold particles.
  - Inlet particle concentration – 2E8/mL (~6 ppb)
  - Total challenge resulted in a fractional filter coverage of 0.2 monolayers

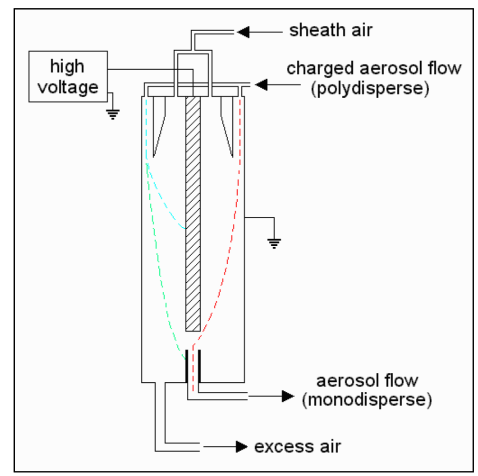
Particle comparison

- Size distributions
- Anticipated capture mechanisms
- "Real worldliness"
- Cost

Liquid Nanoparticle Sizer Description



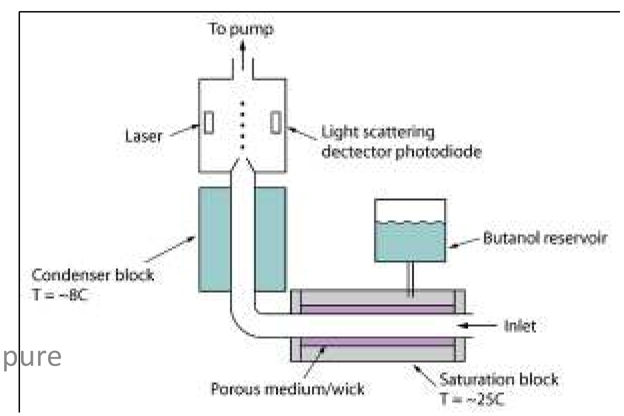
Dynamic Mobility Analyzer (DMA)



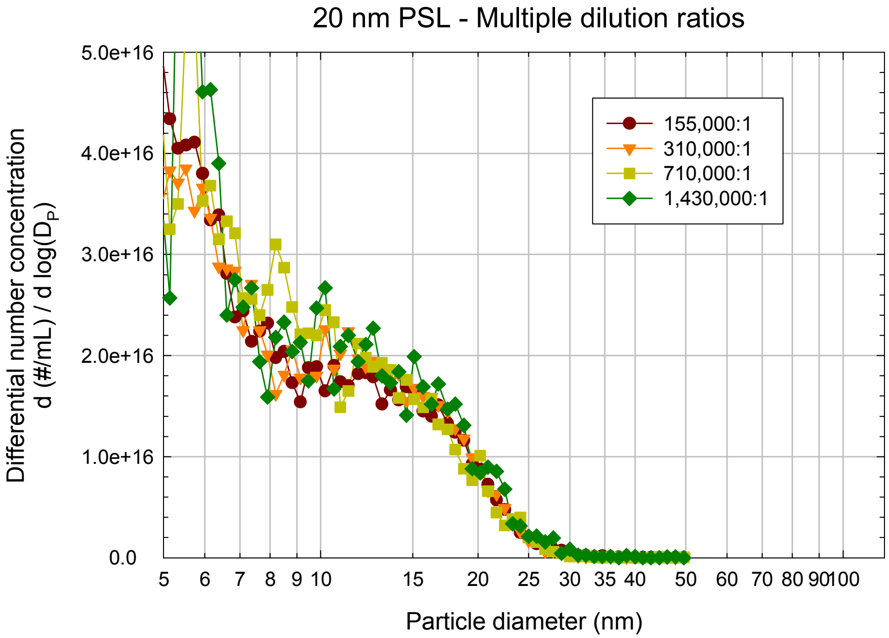
Operating Principle

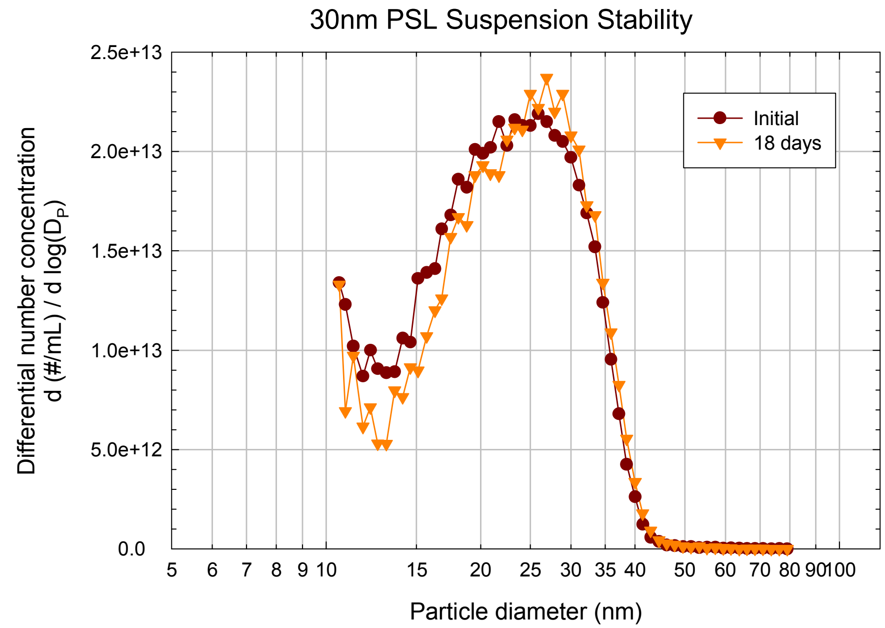
- Nebulizer converts the hydrosol to an aerosol.
- DMA separates particles according to size.
- CPC measures concentrations of particles of each size.

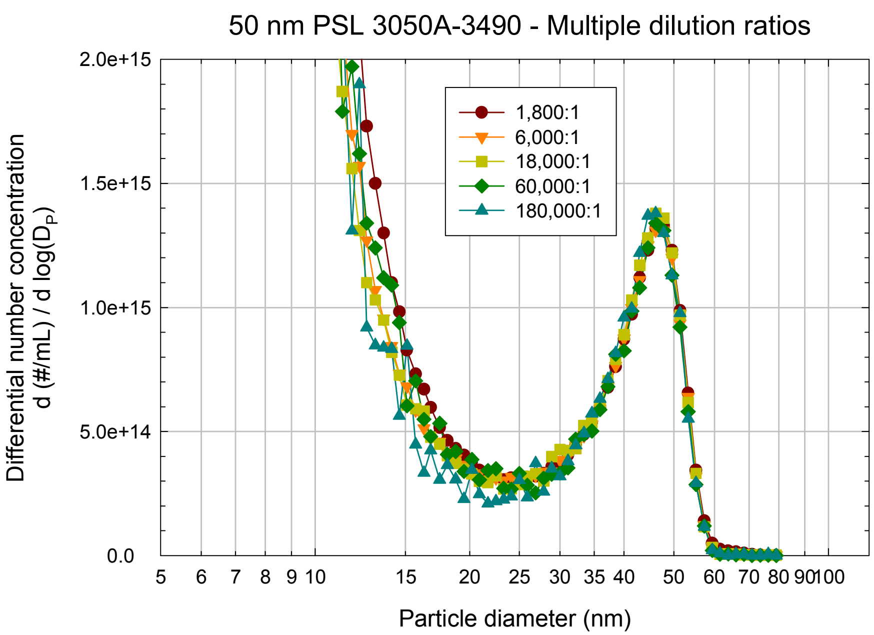
Condensation Particle Counter (CPC)

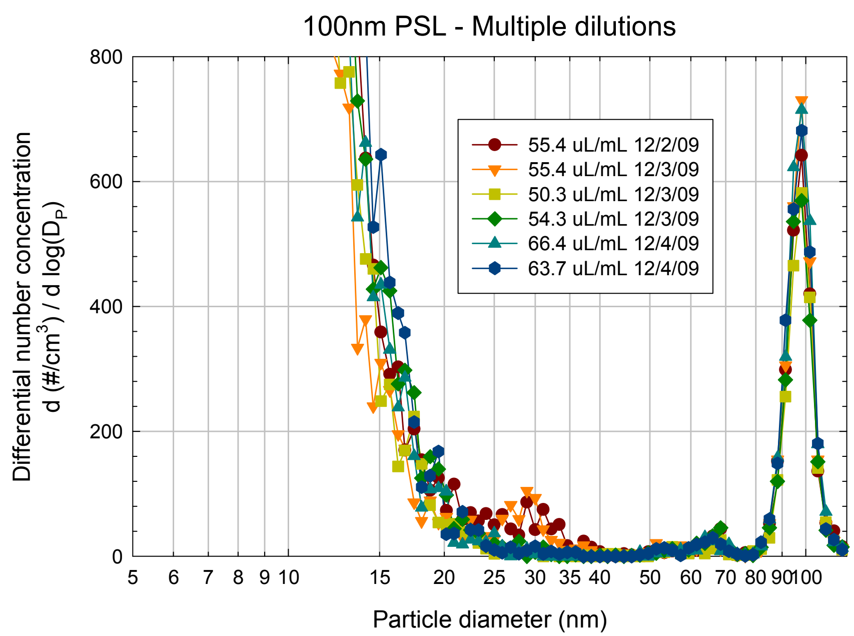


Examples of PSL particle size distributions

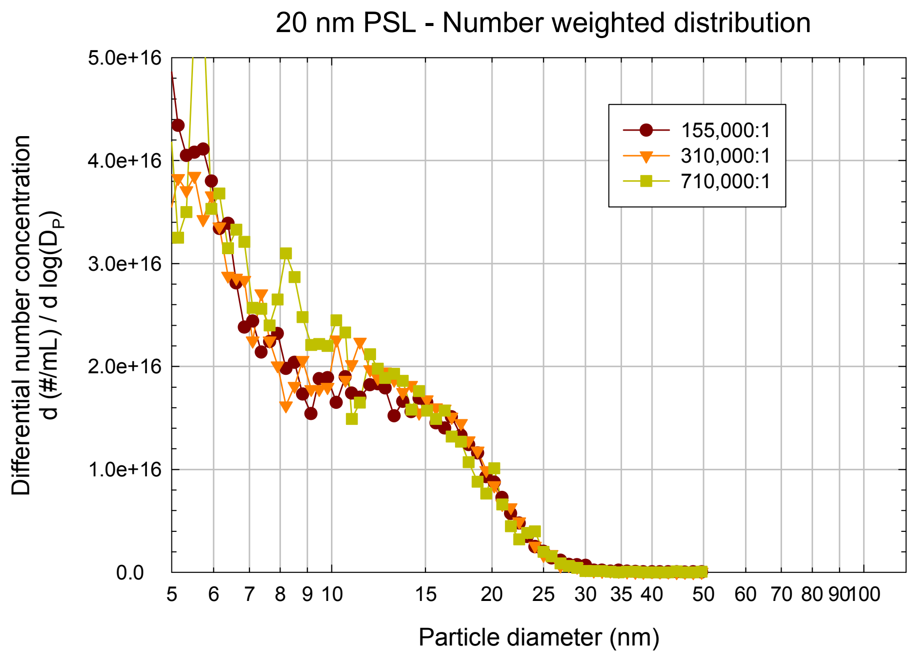


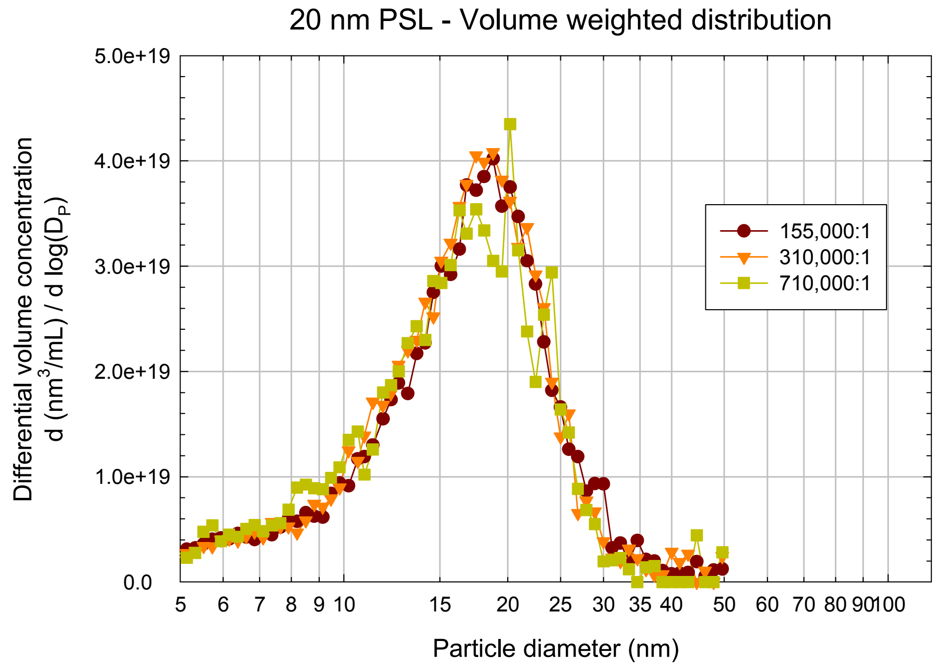




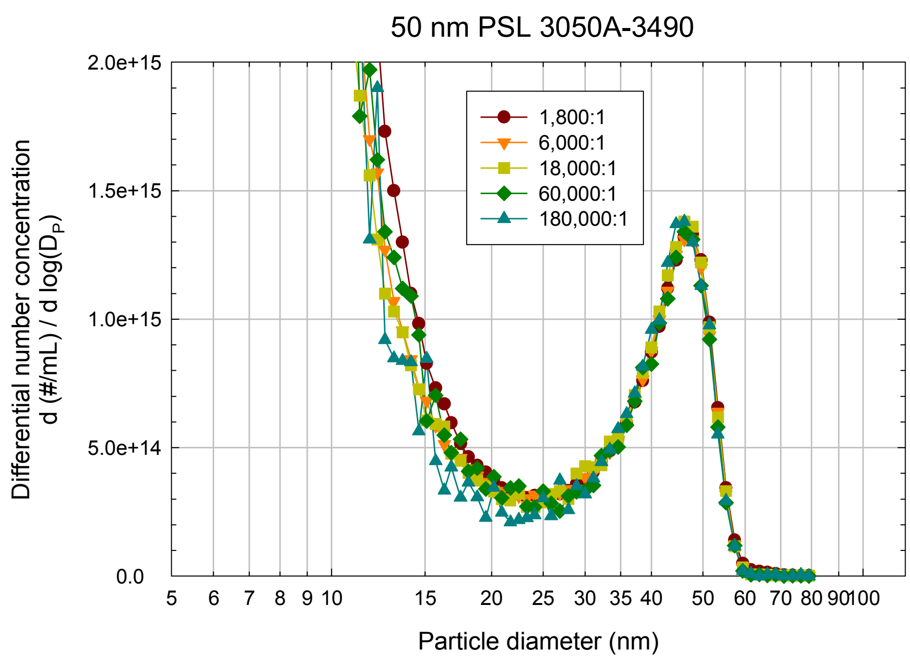


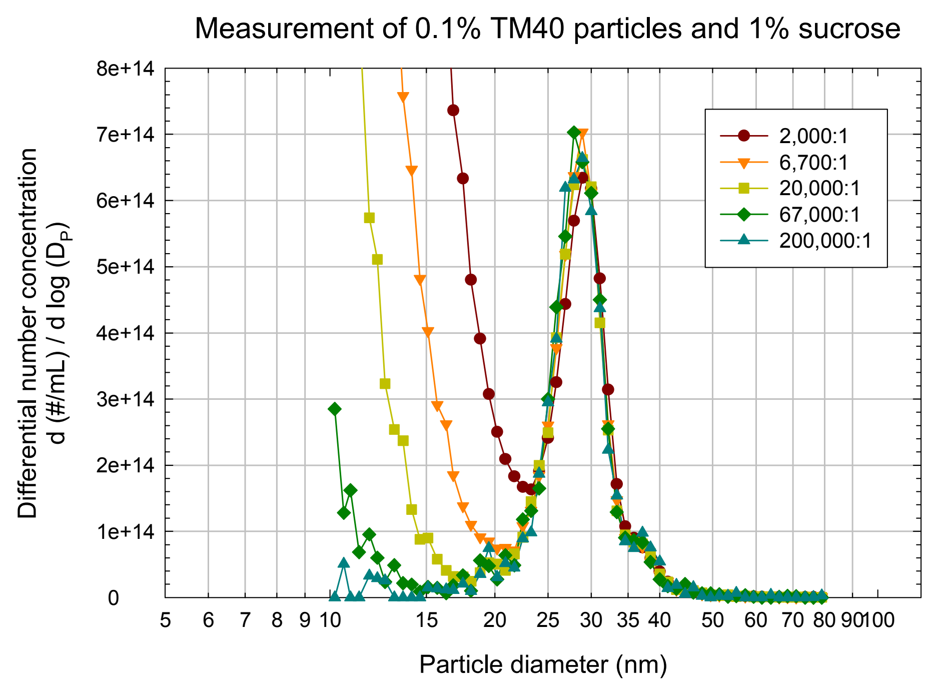
What size are the 20nm PSL particles?????





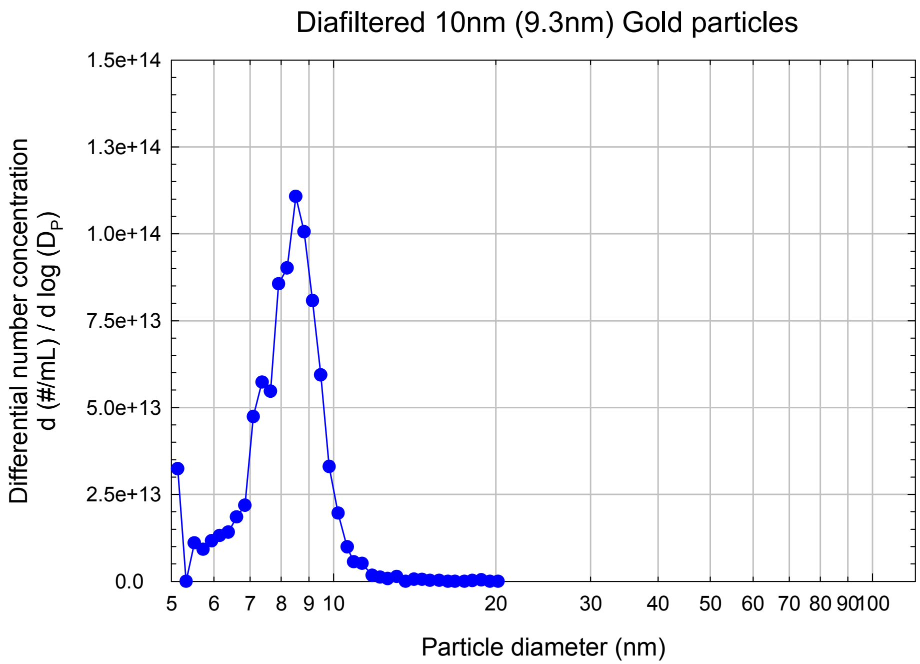
What's the small stuff in the PSL????

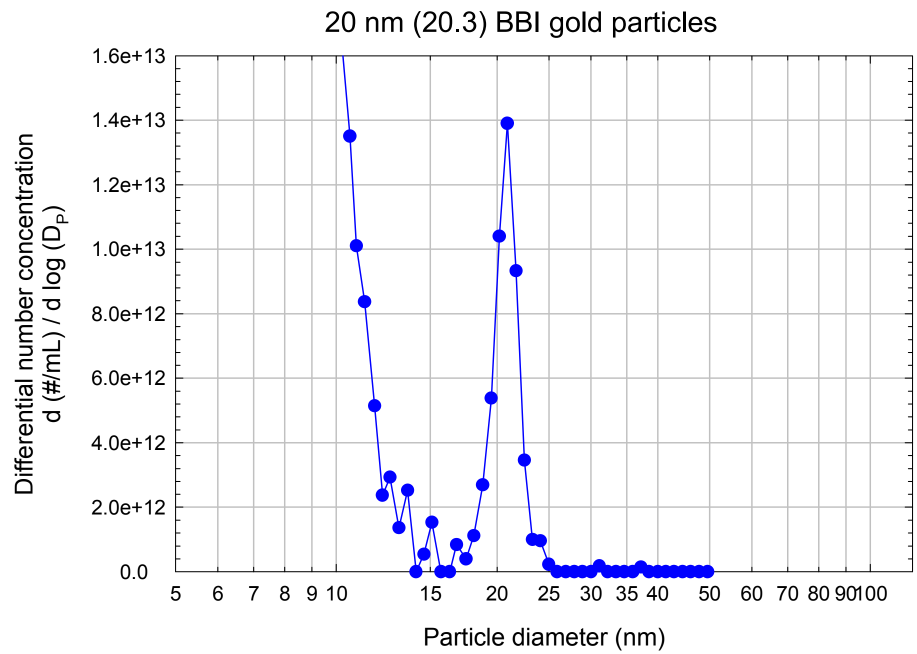


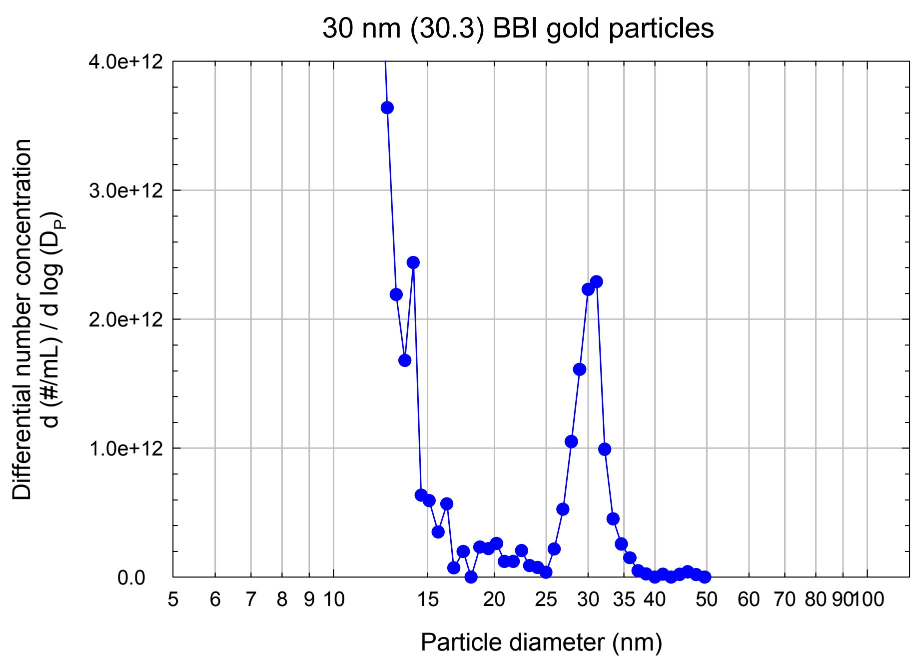


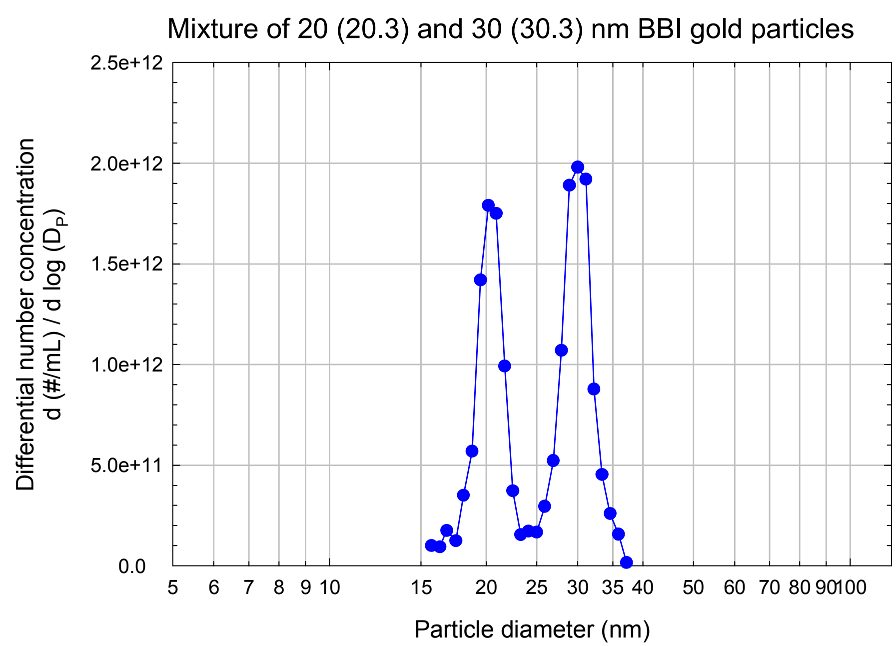
Particles!!!!

Sizing of gold nanoparticles from BBI

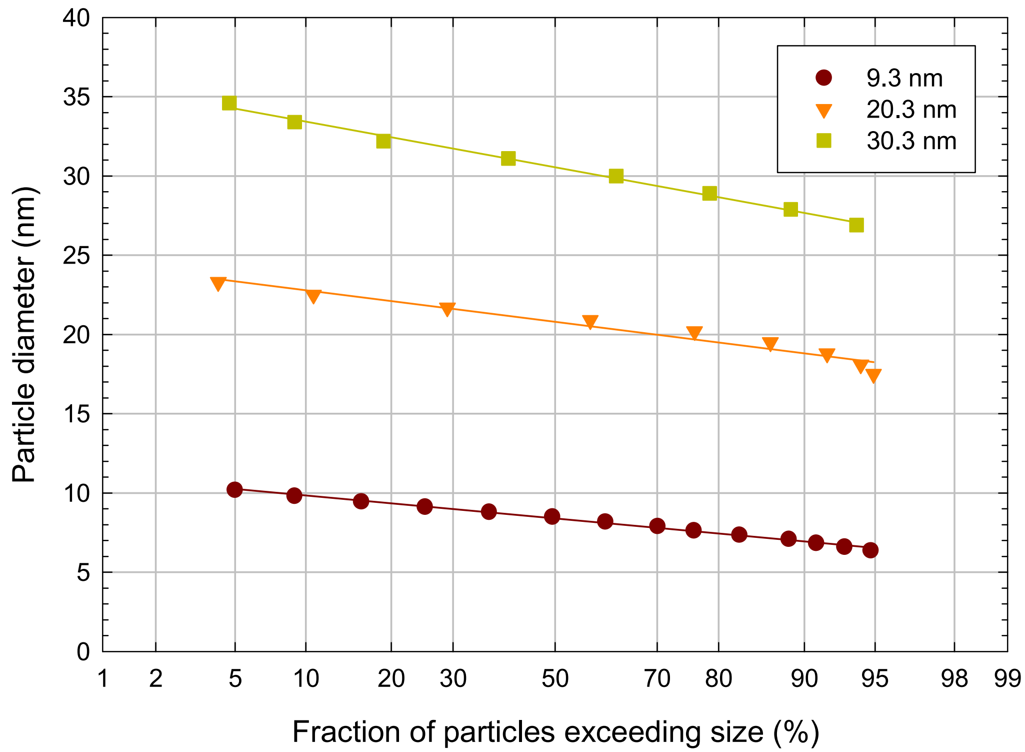








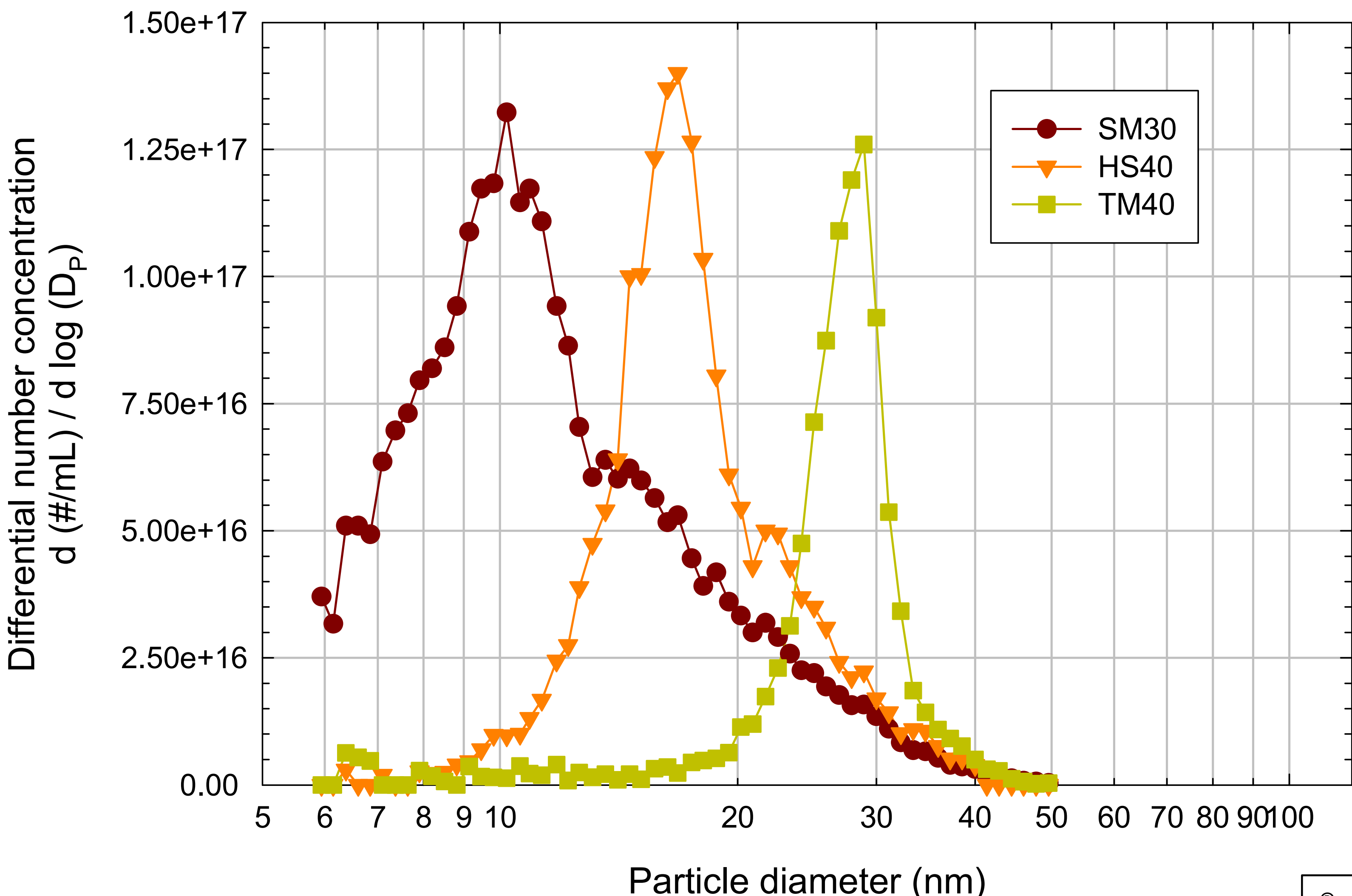
Comparison between claimed and measured gold nanoparticle sizes (from BBI)



Nominal Size (nm)	Claimed size		Measured size	
	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

Don Grant et al, Presented at the 2011 CMP and Ultrapure Conference Sponsored by Levitronix.

Size distributions of Ludox® Colloidal Silica Particles



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Particle size distributions

- Polystyrene latex
  - Available in “mono-dispersed” sizes of 20, 30, 40, 50 nm, etc.
  - Particles smaller than about 50 nm are not very uniform in size.
    - The standard deviation of particle diameters is approximately 6 nm.; regardless of mean size.
    - This results in CVs of ~6% and ~30% for 100 and 20 nm particles; respectively.
  - Suspensions contain high concentrations of small particles.
  - Suspensions contain moderate surfactant concentrations to stabilize the particles.
- Colloidal gold
  - Available in “mono-dispersed” sizes of 5, 10, 15, 20, 25, 30 nm, and larger.
  - The particles are very uniform in size (CV around 8%.)
  - Suspensions contain high concentrations of dissolved species.
- Colloidal silica
  - Available with median sizes of 12, 18, and 28 nm (possibly also smaller sizes).
  - The 28 nm particles are very uniform in size (CV < 10%); the 12nm and 18nm are less uniform.
  - Contain low concentrations of dissolved species.

Particle capture mechanisms

- Particle capture by filters can result from several mechanisms including:
  - Diffusion
  - Interception
  - Impaction
  - Electrostatic attraction
  - Sieving
- Particle capture should be by sieving only
  - Worst case capture mechanism
  - Capture by diffusion, interception, impaction and electostatic attraction and adsorption should be absent (or nearly absent).
  - Desire a strong repulsive force and a weak attractive (Van der Waals) force between the particles and the membrane surface to minimize the potential for adsorption.

Anticipated particle capture mechanisms

- PSL
  - Particle zeta potential (-16 mV) predicts a moderate particle-membrane repulsive force in most cases.
  - Particle composition predicts a large particle-membrane attractive force in most cases.
  - Tests with multiple membrane types indicate significant non-sieving particle capture occurs.
  - Non-sieving capture can be eliminated if surfactant is added to the challenge – not a real-world situation.
- Colloidal gold
  - Particle zeta potential (-11 mV) predicts a low to moderate particle-membrane repulsive force in most cases.
  - Particle composition predicts a moderate particle-membrane attractive force in most cases.
  - The solution in which the purchased particles are suspended contains a high concentration of dissolved conductive material.
  - Tests with multiple membrane types indicate significant non-sieving particle capture occurs.
  - Non-sieving capture can be reduced/eliminated by modifying the surface of the particles.
- Colloidal silica
  - Particle zeta potential (-16 mV) predicts a moderate particle-membrane repulsive force in most cases.
  - Particle composition predicts a weak particle-membrane attractive force in most cases.
  - Tests with multiple membrane types indicate little if any non-sieving particle capture.

Particle "real-worldliness"

- PSL
  - The particles are plastic spheres and are not believed to be representative of particles in UPW systems.
- Colloidal gold
  - The particles are not believed to be representative of particles in UPW systems.
- Colloidal silica
  - UPW systems are known to contain colloidal silica.

Particle comparison

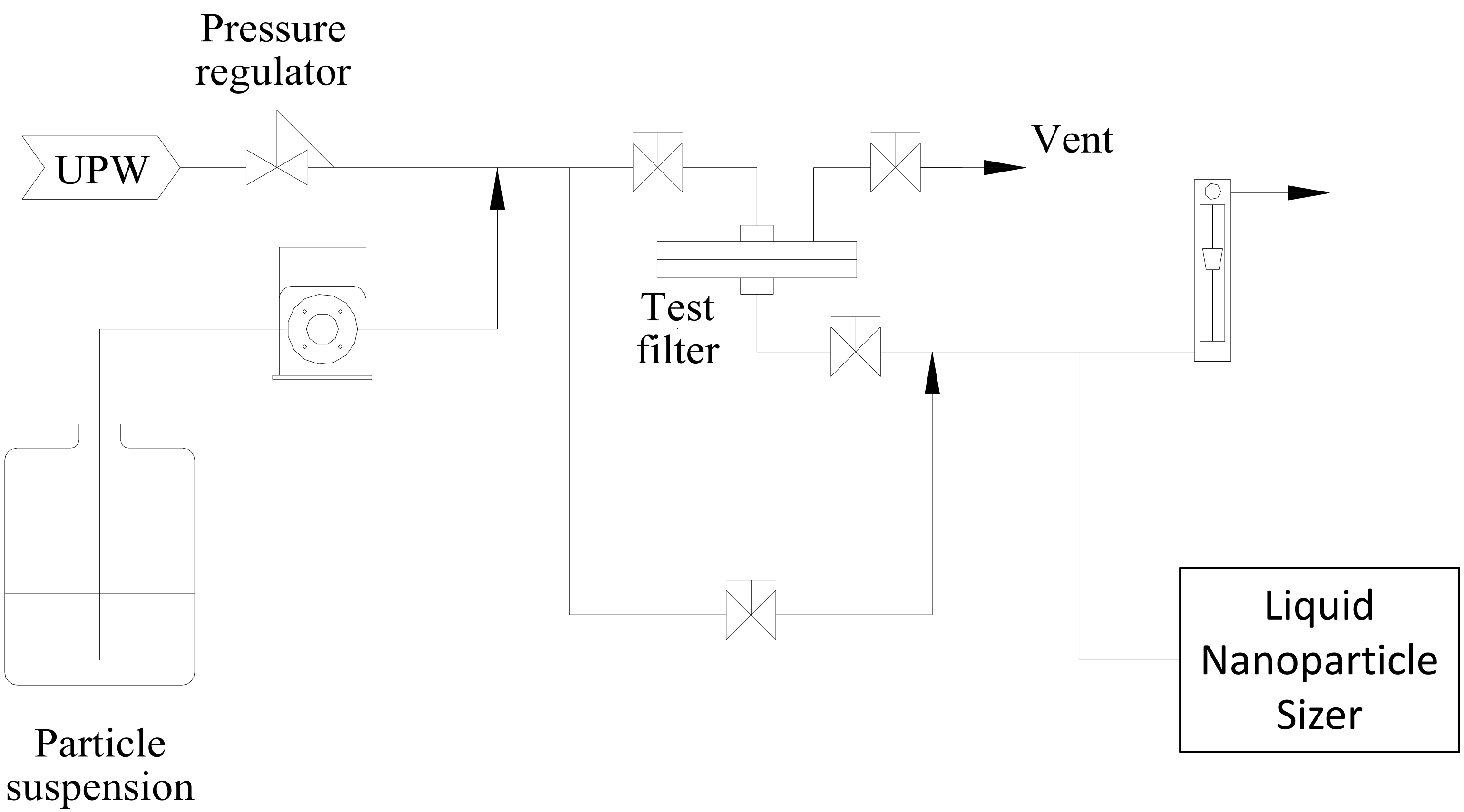
Particle Type	Sizes Available	"Real World"?	Sieving only?	Cost of particles per gram
PSL	Yes	No	Can be achieved by adding surfactant.	$1,800
Colloidal Gold	Yes	No	Can be achieved by surface modification.	$23,000
Colloidal Silica	Yes	Yes	Yes	$0.08

Colloidal silica appears to be the best choice.

Filter cartridge testing

- Cartridges were challenged with 3 types of 30 nm particles (PSL, colloidal gold, colloidal silica)
- Three separate cartridges were tested.
- Each cartridge was challenged with multiple particle types. The challenge order was varied amongst the cartridges.
- One cartridge was also challenged with a mixture of 15 nm colloidal gold and 30 nm colloidal silica particles.

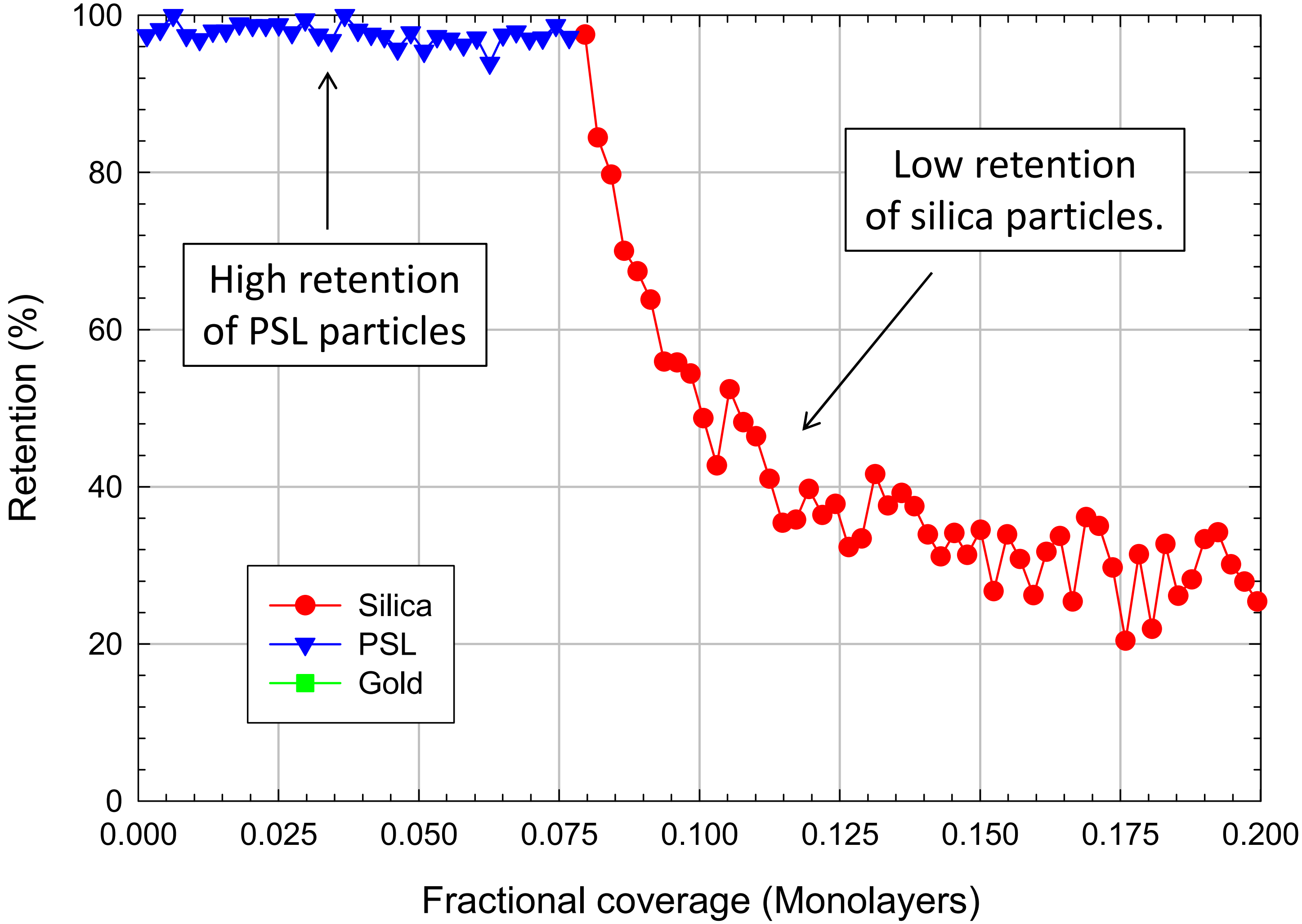
Filter test system schematic



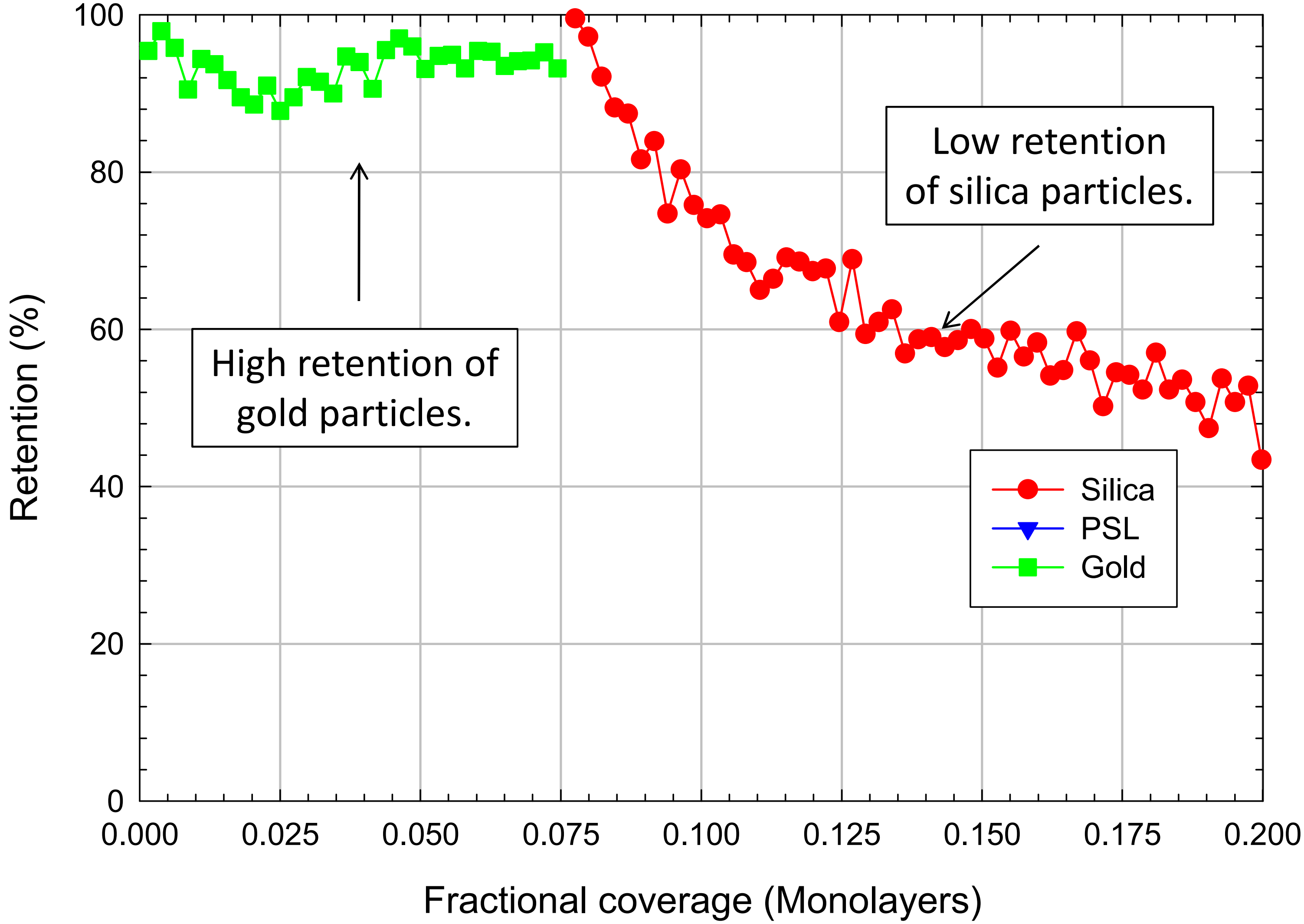
Test Procedure

- The cartridges was flushed until the filtrate approached the system background concentration (10^6/mL > 10 nm).
- The filter was challenged with 2E8 particles/mL (~6 ppb).
- The challenge concentration was verified.
- The face velocity throughout the test was 0.11 cm/min.

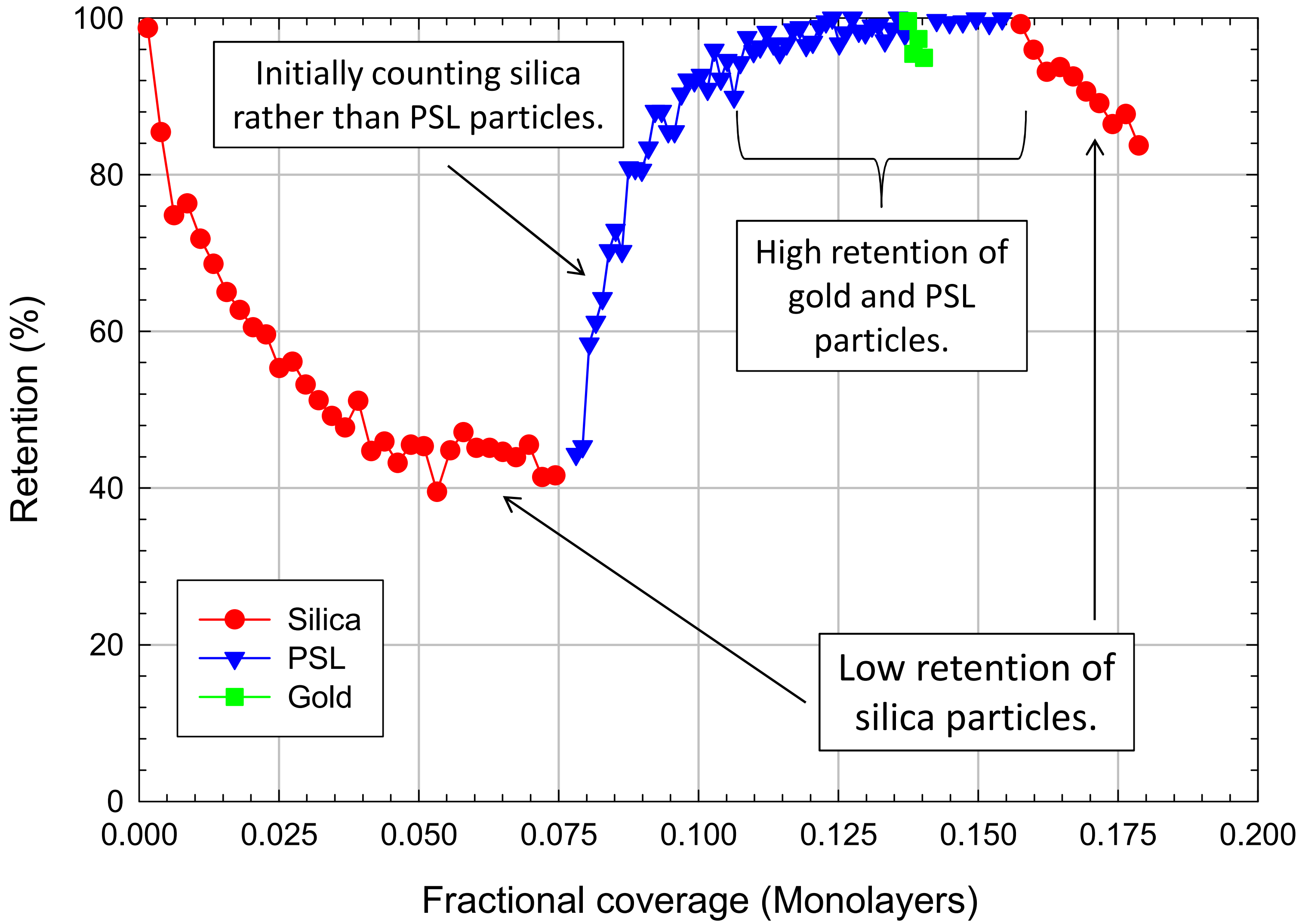
Retention of PSL followed by silica
(Cartridge #1)



Retention of gold followed by silica
(Cartridge #2)

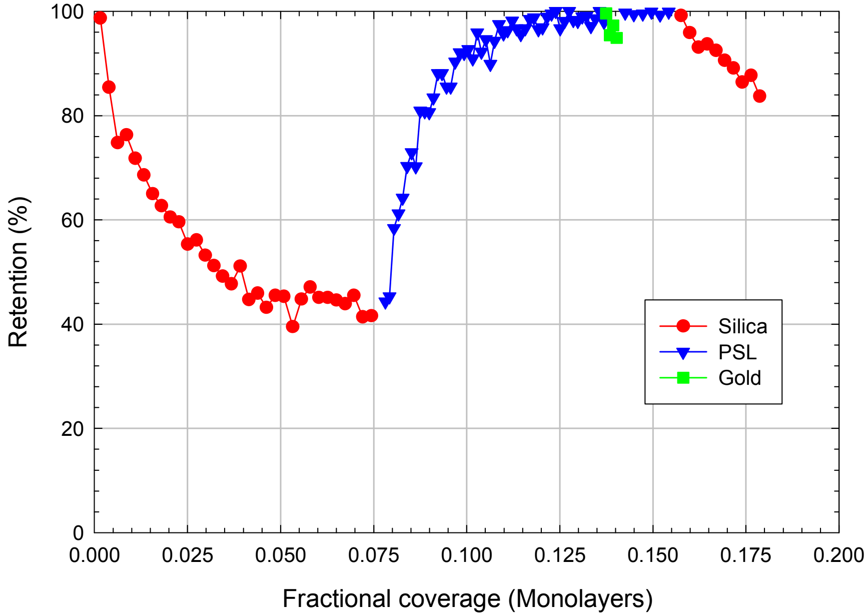


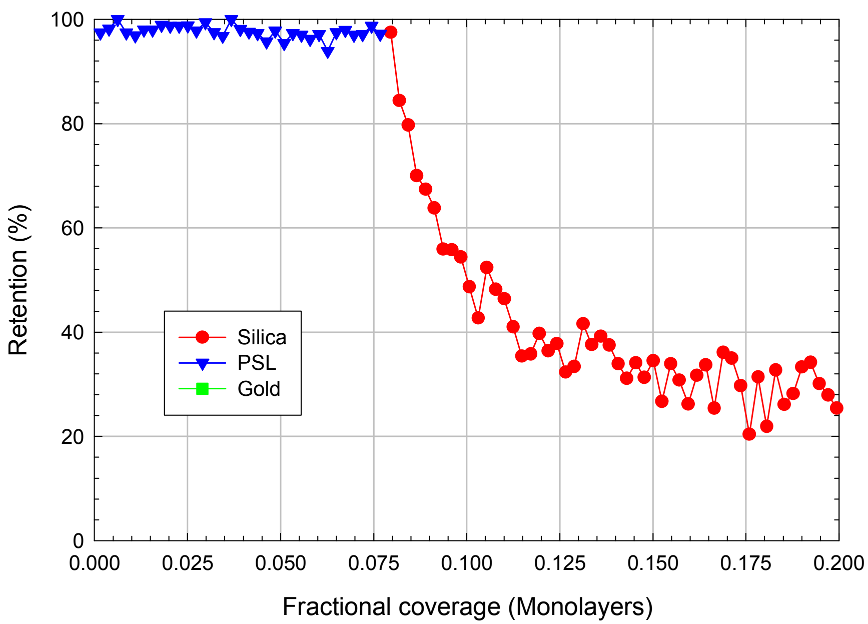
Retention of multiple particle types
(Cartridge #3)

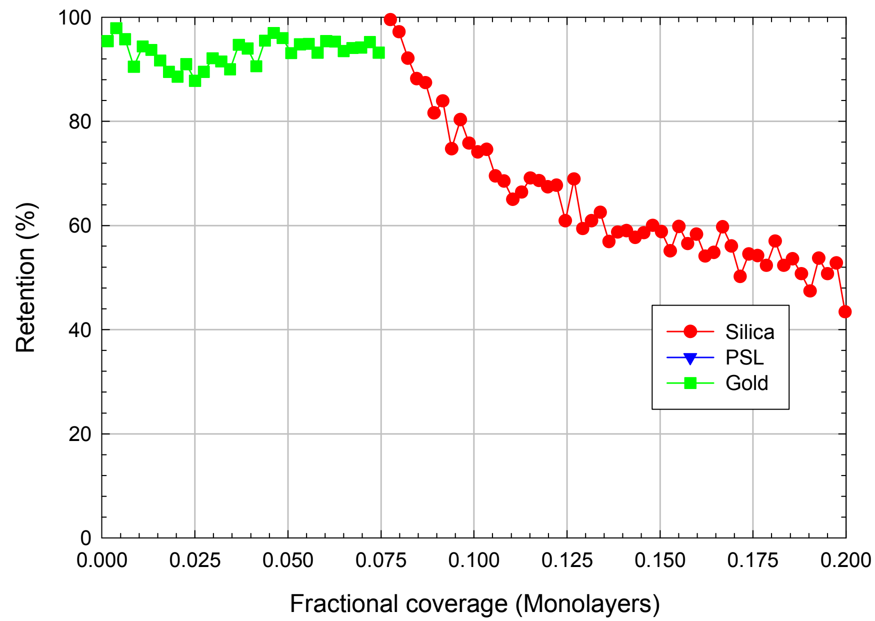


Retention of different 30nm particles types by a commercially available UPW filter cartridges

- 3 separate filters were tested.
- Each was tested with a sequence of particle types.
- In all cases the challenge concentration was 2E8/mL.

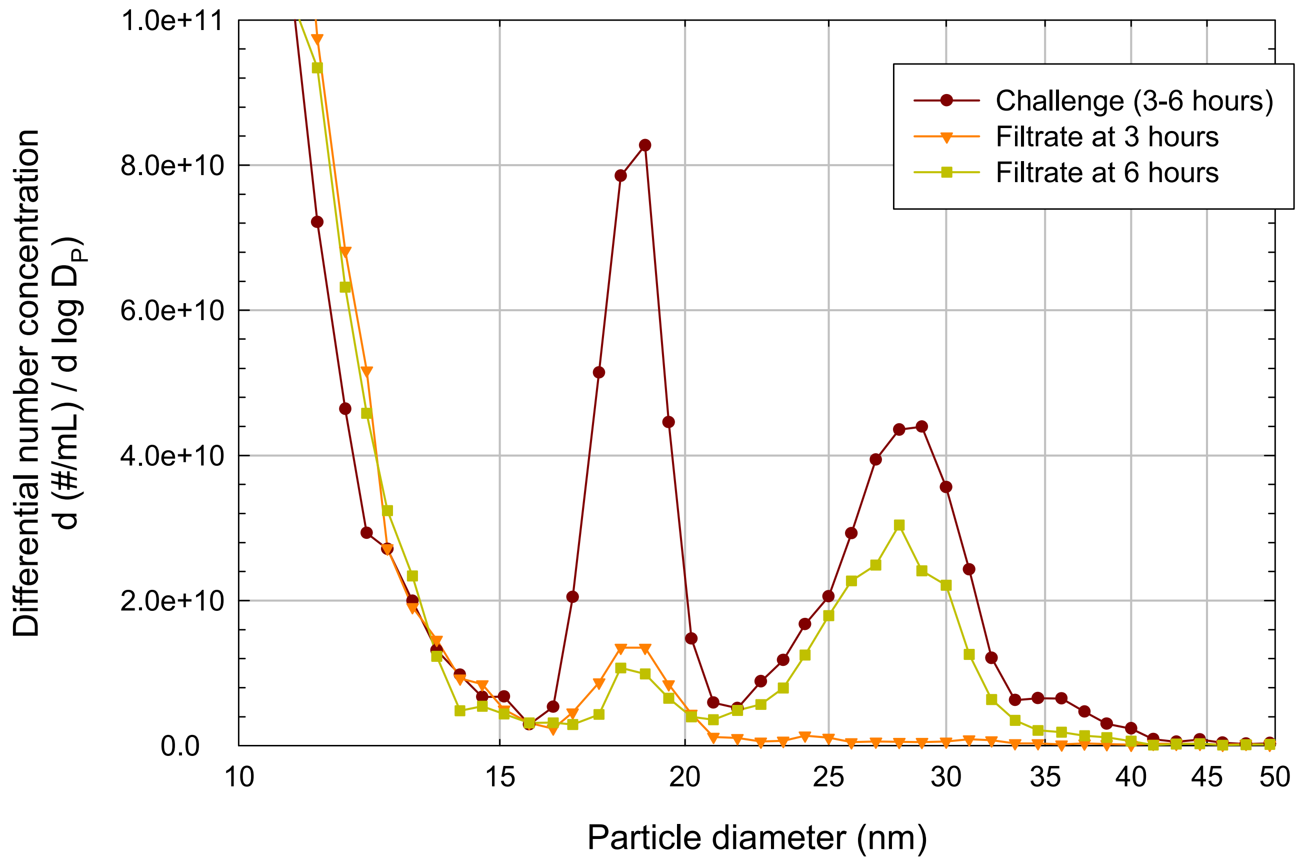




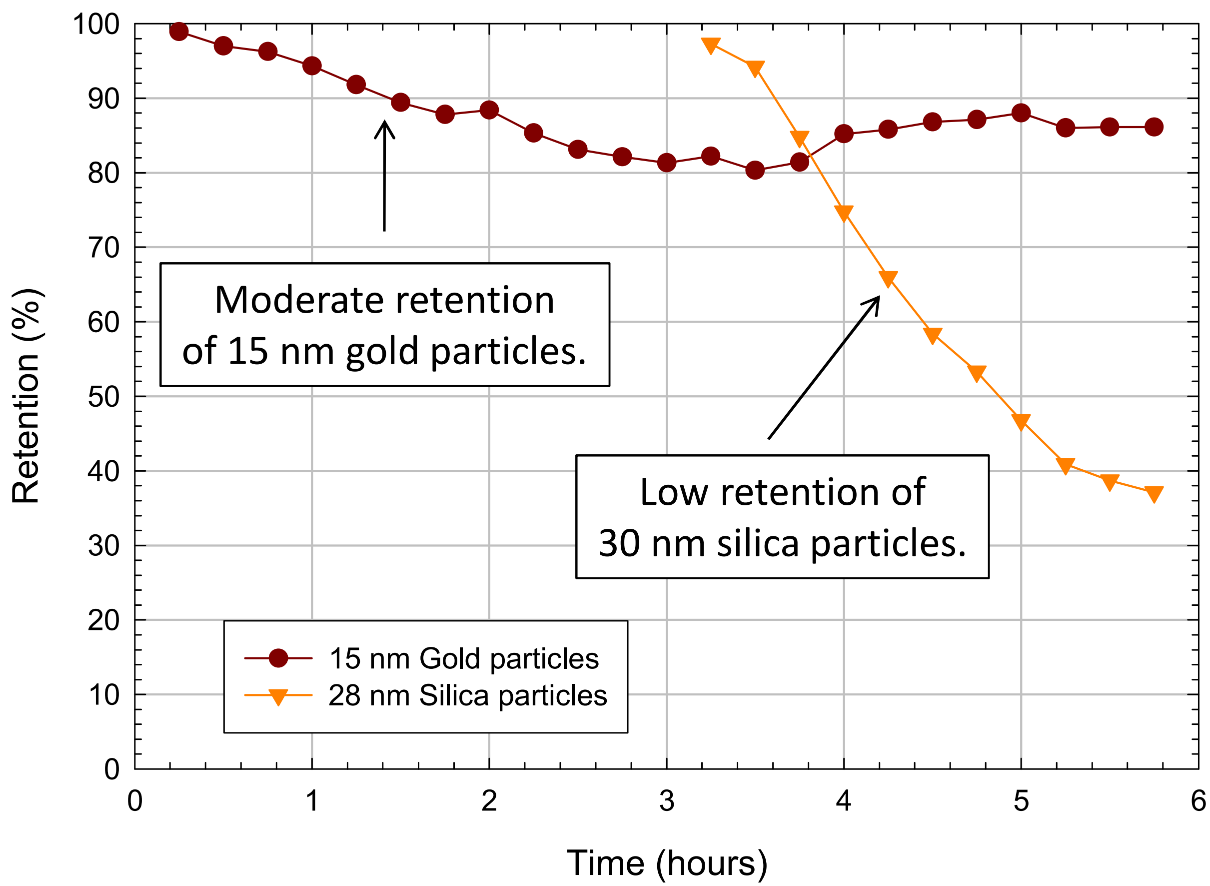


Retention of a mixture of 15 nm gold and 30 nm silica by a commercially available UPW filter

- Challenged with 3E8/mL gold particles only for first 3 hours
- Then 3E8/mL gold + 3E8/mL silica particles for next 3 hours.



Retention of a mixture of 15 nm gold and 30 nm silica by a commercially available UPW filter



- Challenged with 3E9/mL gold particles only for first 3 hours.
- Then 3E9/mL gold + 3E9/mL silica particles for next 3 hours.

Particle retention comparison

- Retention of PSL and gold particles was significantly higher than silica particles.
- Retention of silica particles is predominately by sieving while PSL and gold particles are likely removed by several capture mechanisms.
- Silica is the preferred particle type for UPW filter retention testing.

Summary and conclusions

- Test methods to measure the retention of sub-50nm particles by UPW filters are needed.
- The methods should:
  - Use real-world particles that are removed by sieving ,
  - Test the filter under representative conditions of particle concentration, particle loading and face velocity
  - Be applicable to cartridges as well as filter samples.
  - Not be cost prohibitive.
- Filter retention tests performed with PSL, gold, and silica 30 nm particles indicated that silica is the preferred challenge particle.