# The effect of particle composition on filter removal of sub-30nm particles from UPW

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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 this size can reduce finished device yield and reliability.
- Particles in UPW that contacts wafer surfaces during processing can deposit on the wafer surface so microfilters and ultrafilters are used to remove the particles.
- Test methods are needed to measure filter particle removal efficiency of particles as small as 5-15 nm.
- Testing should be performed under “worst case” conditions to ensure adequate filter performance in “real life” conditions.
- Particle chemical composition can have a significant effect on filter particle capture efficiency.

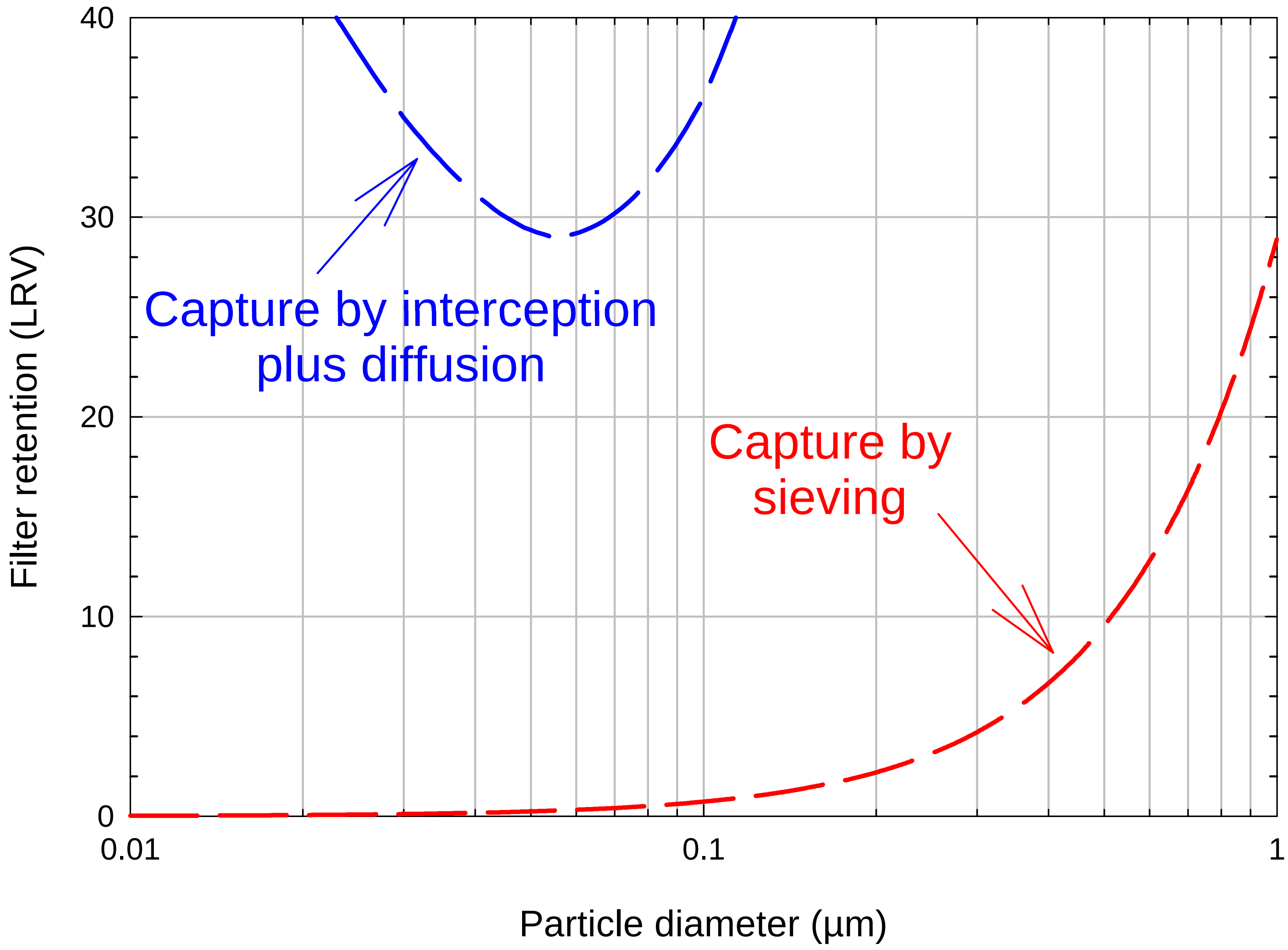
# Outline

- Introduction
- Particle capture mechanisms
  - Description
  - Which are operative????
- Comparison of candidate challenge particle properties
- Examples of retention of candidate test particles by a 30 nm UPW filter
- Summary and conclusions

# Particle capture mechanisms

- Particle capture by filters can result from multiple mechanisms including:
  - Diffusion
  - Interception
  - Impaction
  - Electrostatic attraction
  - Sieving

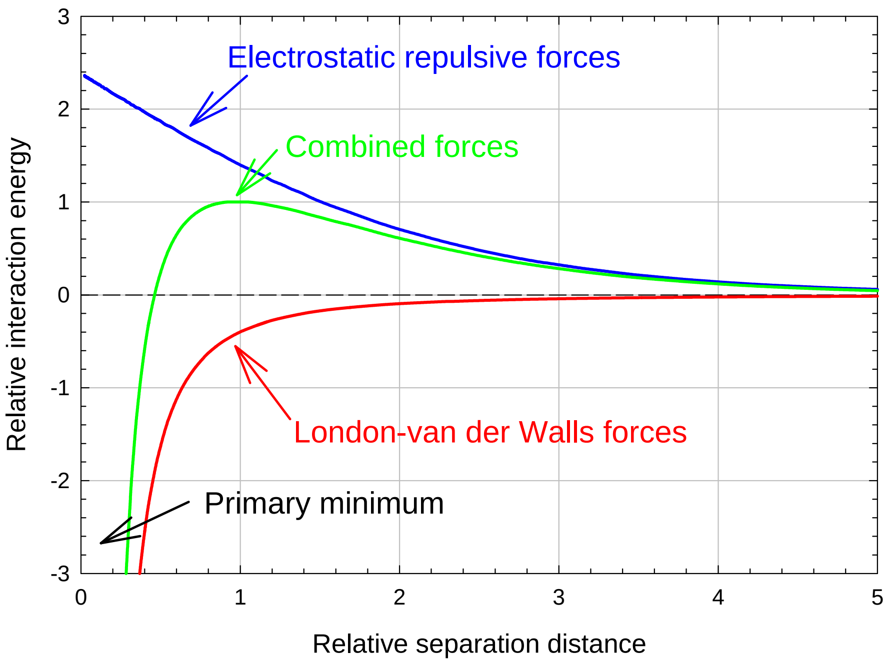
# Removal of particles from UPW by a 0.2 µm rated filter by different capture mechanisms

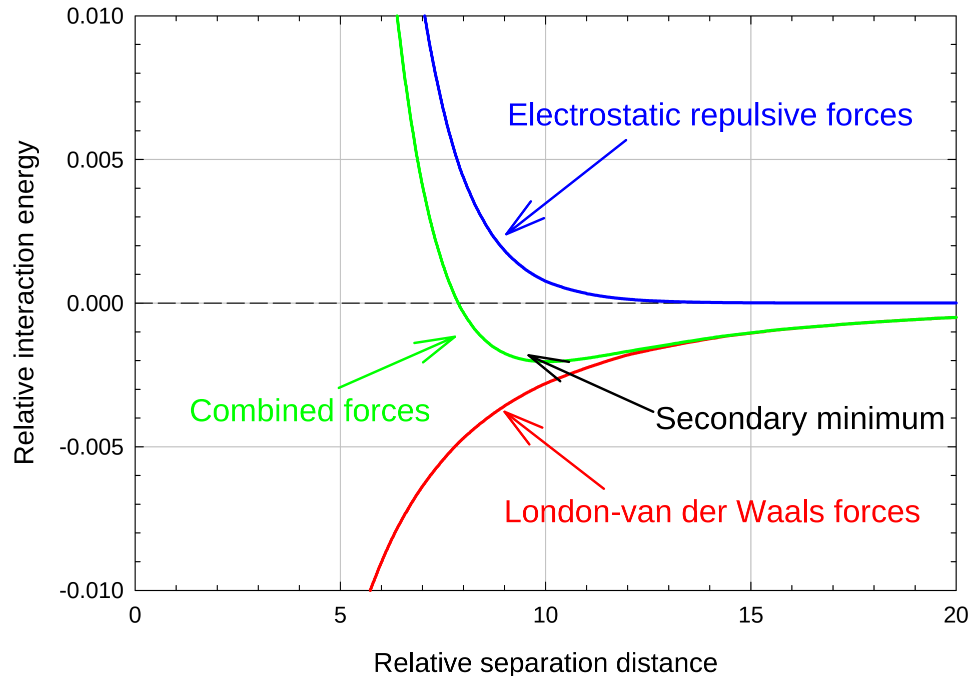


# What controls which capture mechanisms are active???????????

- DLVO theory (Deryaguin, Landau, Verwey and Overbeek)
- Describes the interaction between surfaces
  - Electrostatic forces
  - Forces between atoms and molecules (London-van der Walls forces)
- London-van der Walls forces are short range and generally attractive
- Electrostatic forces are long range, can be attractive or repulsive, but are most often repulsive.

# DVLO Theory
## (assuming repulsive electrostatic forces)



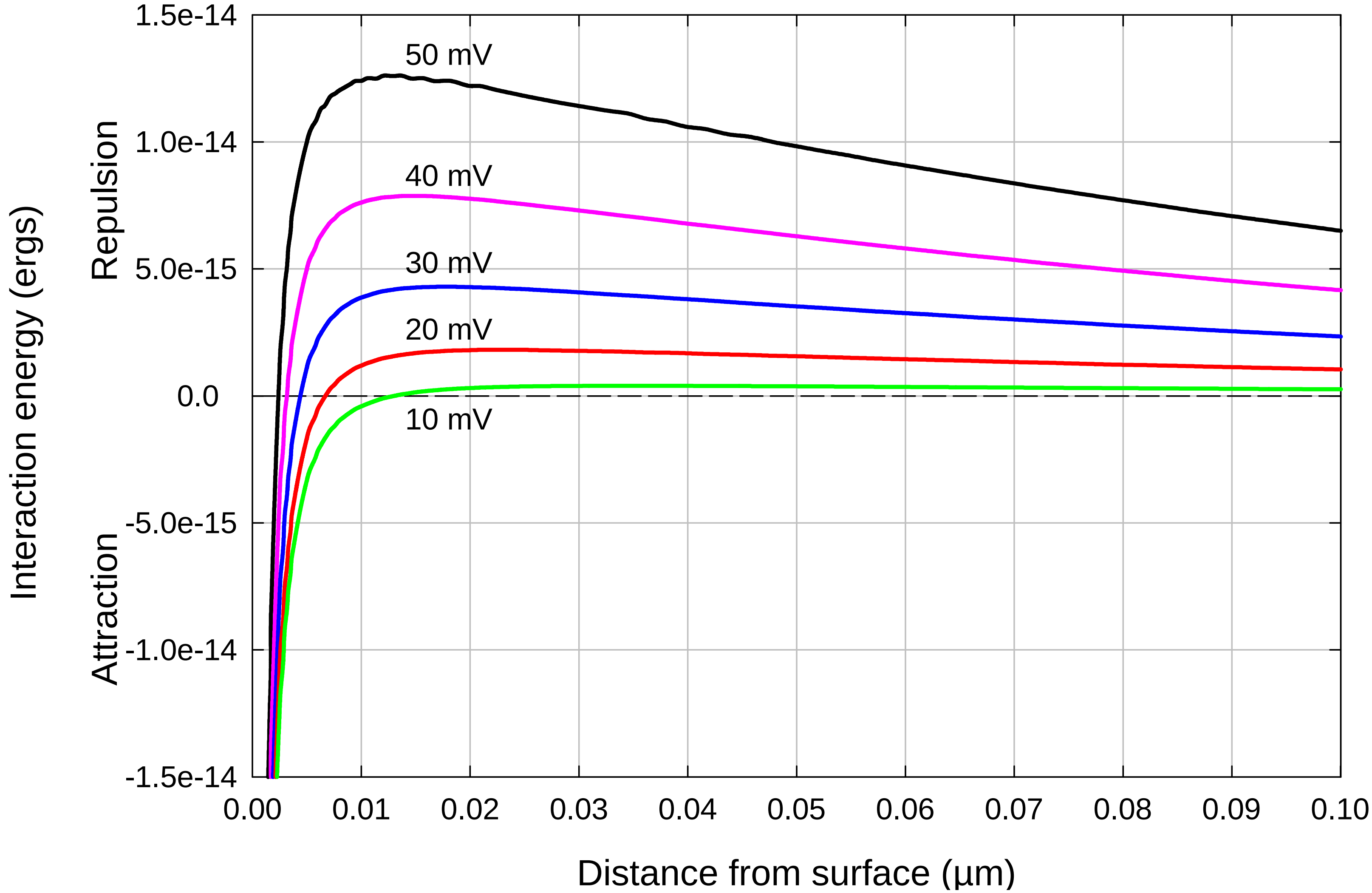



# Particle capture mechanisms

- 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 (London-van der Waals) force between the particles and the membrane surface to minimize the potential for adsorption.

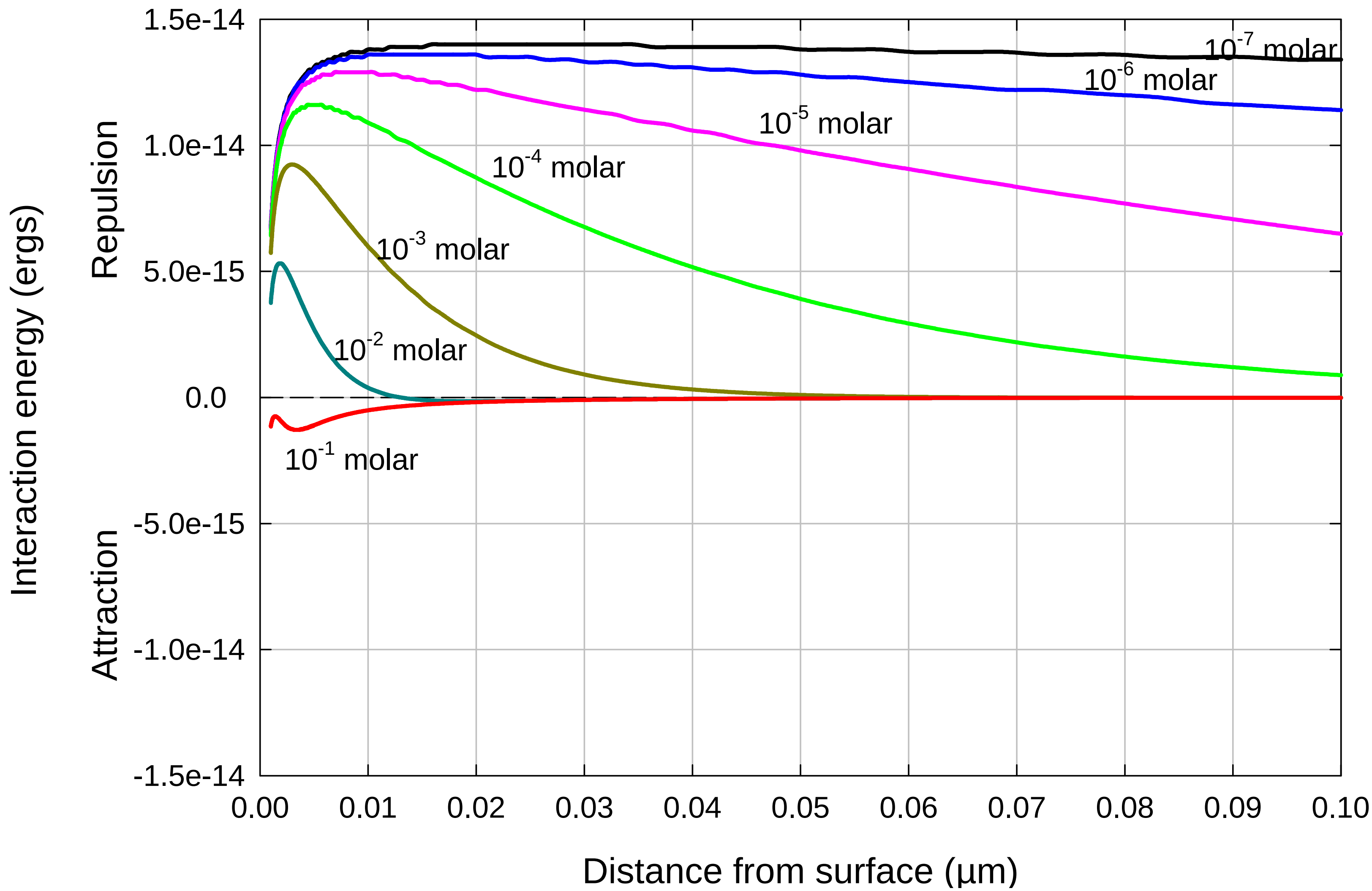
# The effect of surface charge on interaction energy

Hamaker constant $10^{-13}$ ergs, $10^{-7}$ moles/liter, 50 nm particles



# The effect of ionic strength on interaction energy

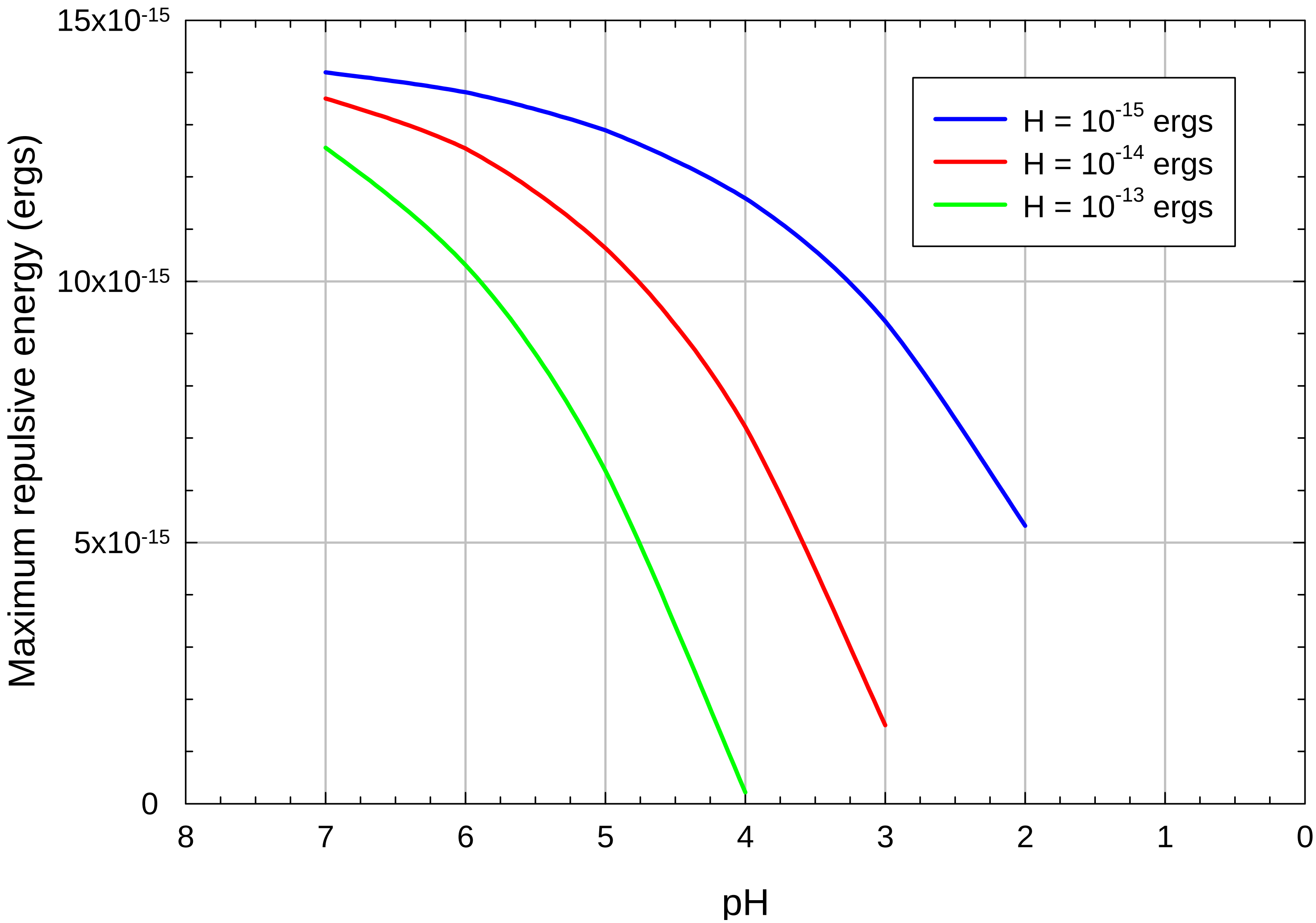
Hamaker constant = $10^{-15}$ ergs, surface charge 50 mv, 50 nm particles




DVLO Predictions Apr 2011

# The effect of the Hamaker constant (H) on the maximum repulsive energy

Surface charge 50 mv, 50 nm particles



# Conditions to minimize adsorption

- High repulsive electrostatic force.
- Weak attractive force.
- Low ionic strength.

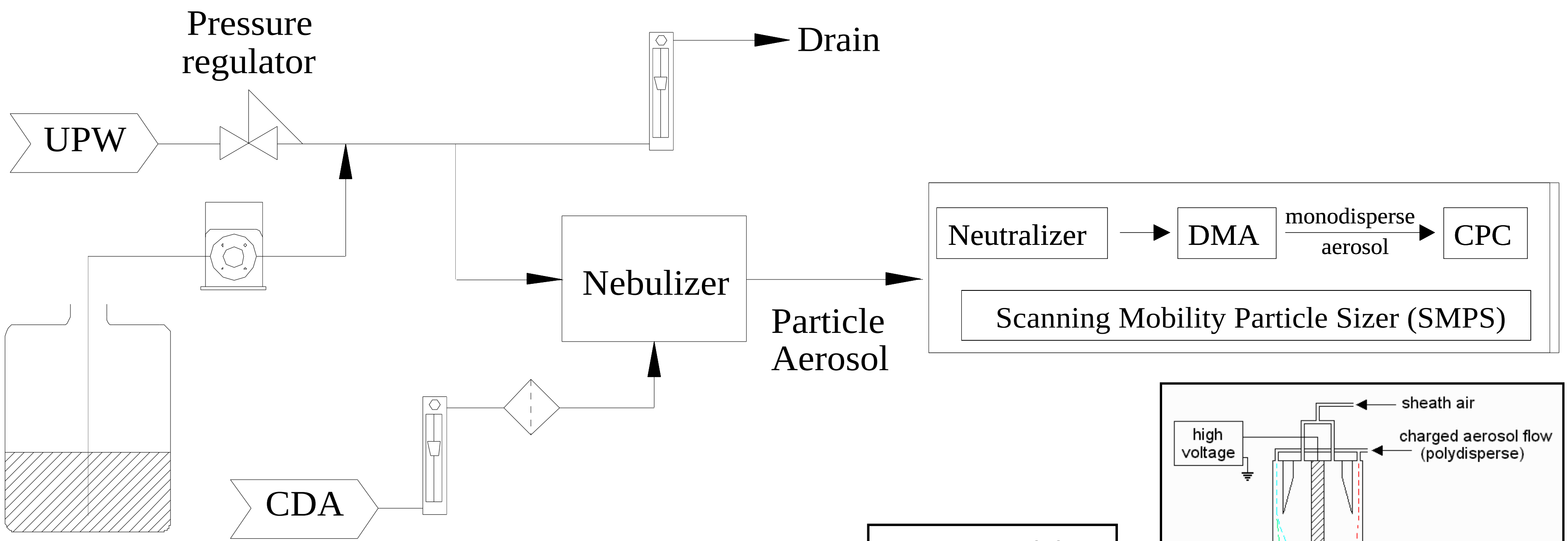
# This study

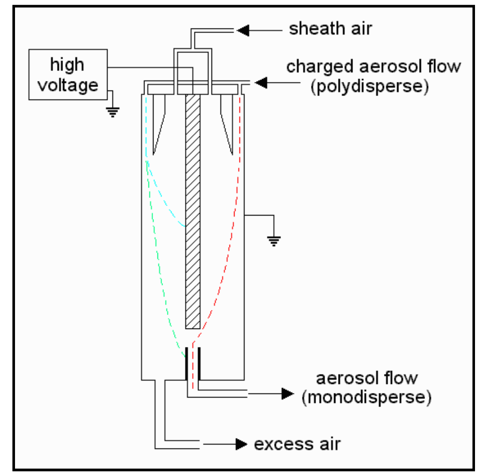
- Focused on measuring the effect of particle type on filter retention.
- Measured the retention of different types of particles by a 30 nm commercially-available UPW filter cartridge.
- Particle types evaluated
  - Polystyrene latex (PSL)
  - Colloidal gold
  - Colloidal silica
- Test conditions employed
  - 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 – $2x10^8$/mL (~6 ppb)
  - Total challenge resulted in a fractional filter coverage of 0.2 monolayers based on particle cross-sectional area

# Particle comparison

- Size distributions
- Anticipated capture mechanisms
- “Real worldliness”
- Cost

# Liquid Nanoparticle Sizer Description

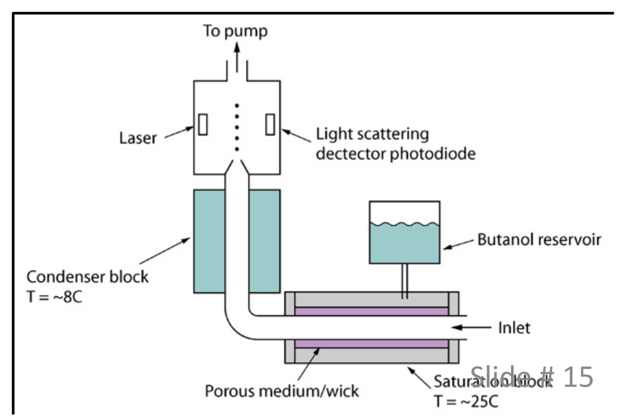



**Dynamic Mobility Analyzer (DMA)**

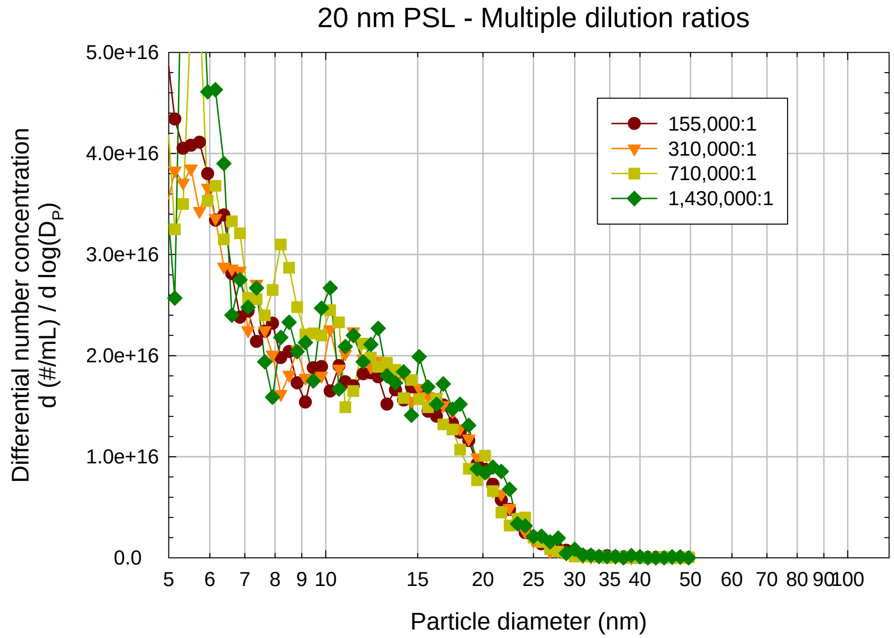
**Operating Principle**

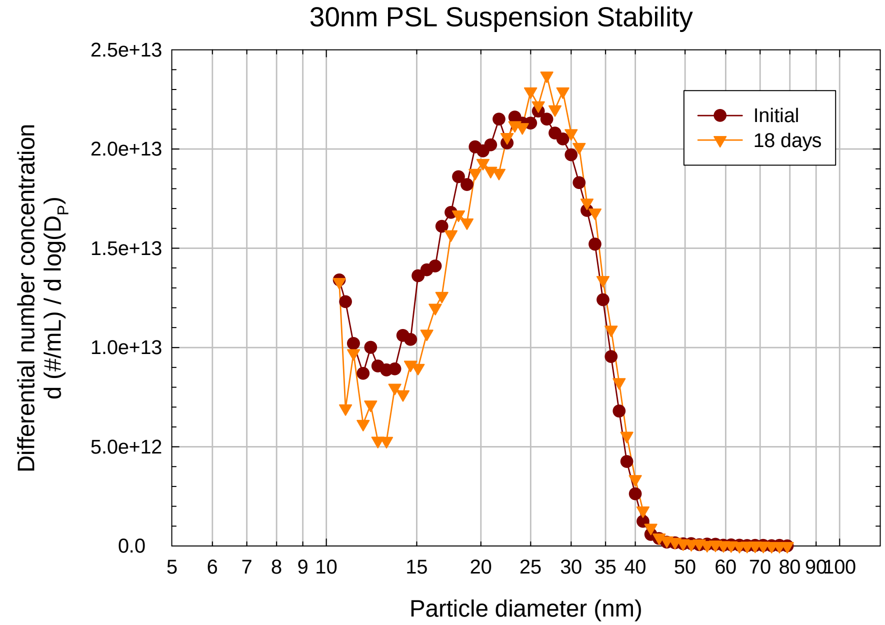
- Nebulizer converts the hydrosol to an aerosol.
- DMA separates particles according to size.
- CPC measures concentrations of particles of each size.

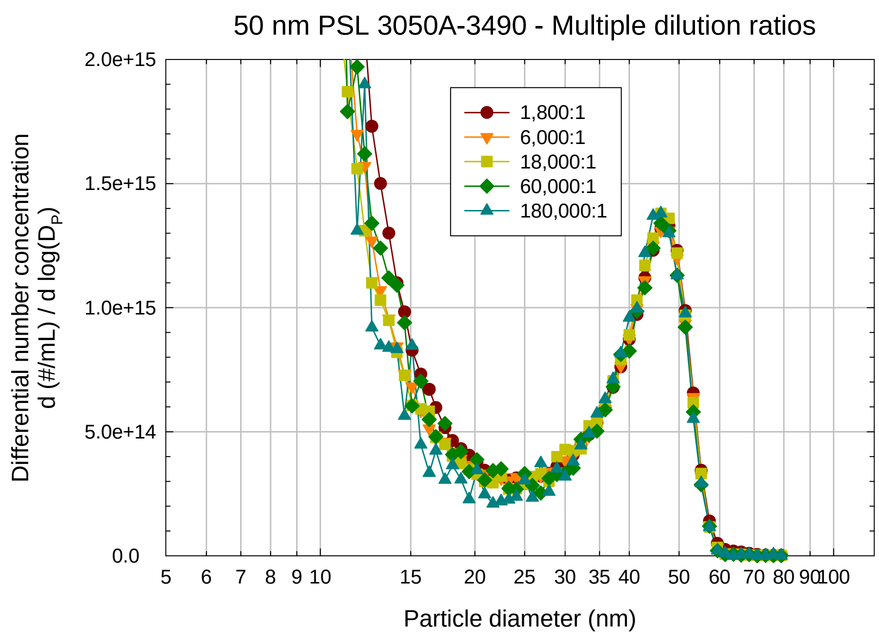


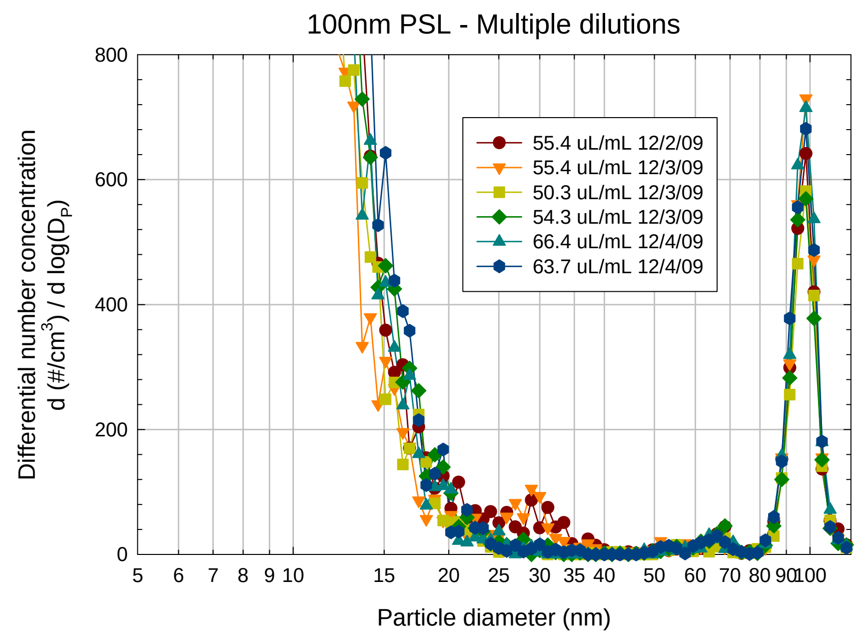

**Condensation Particle Counter (CPC)**

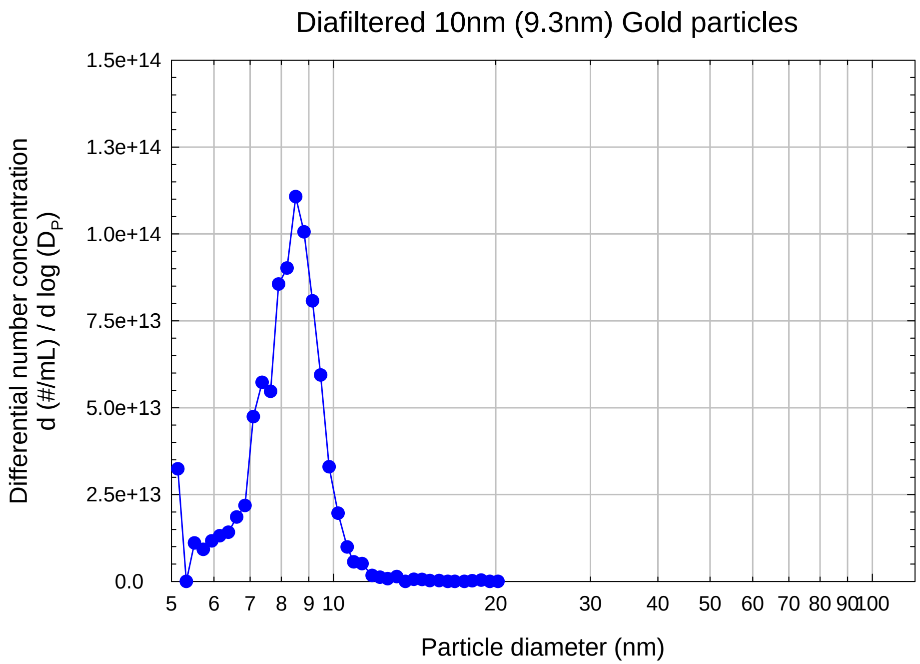
# Examples of PSL particle size distributions

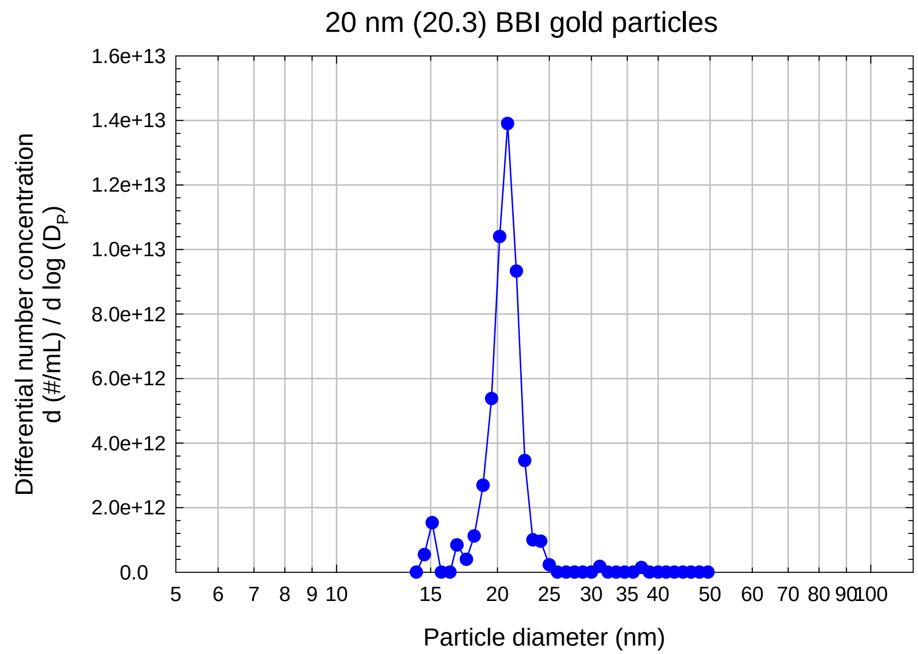


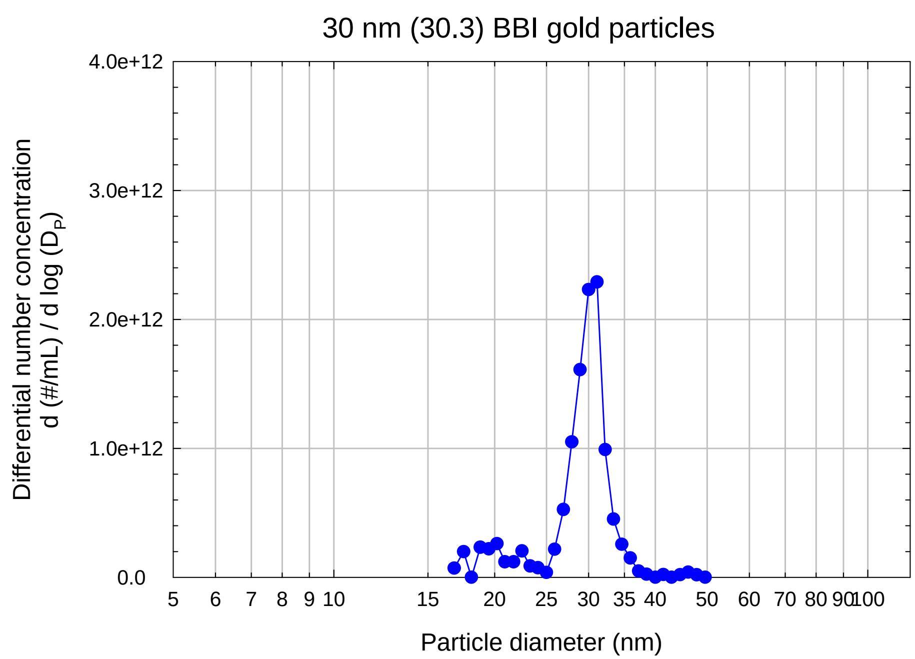



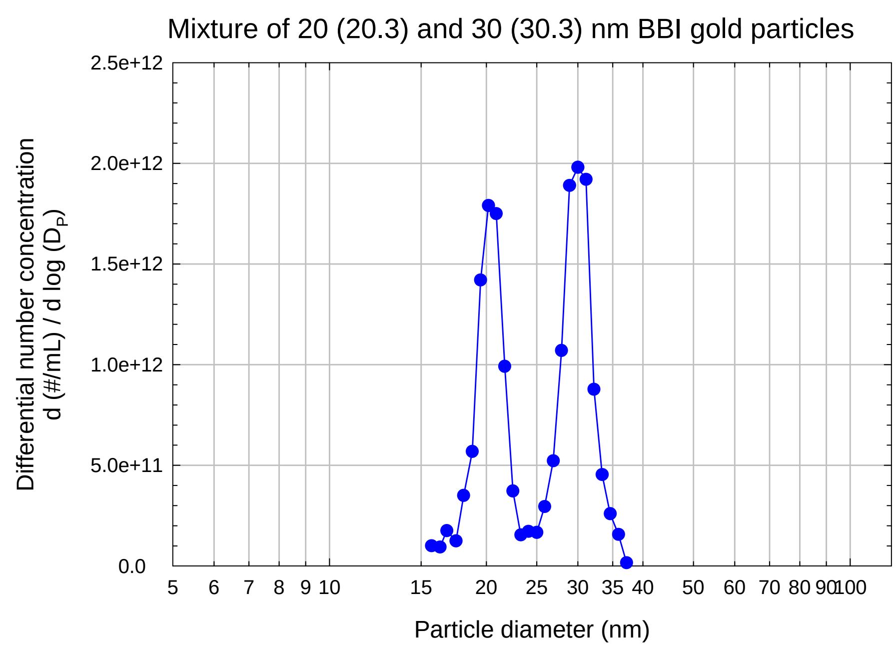





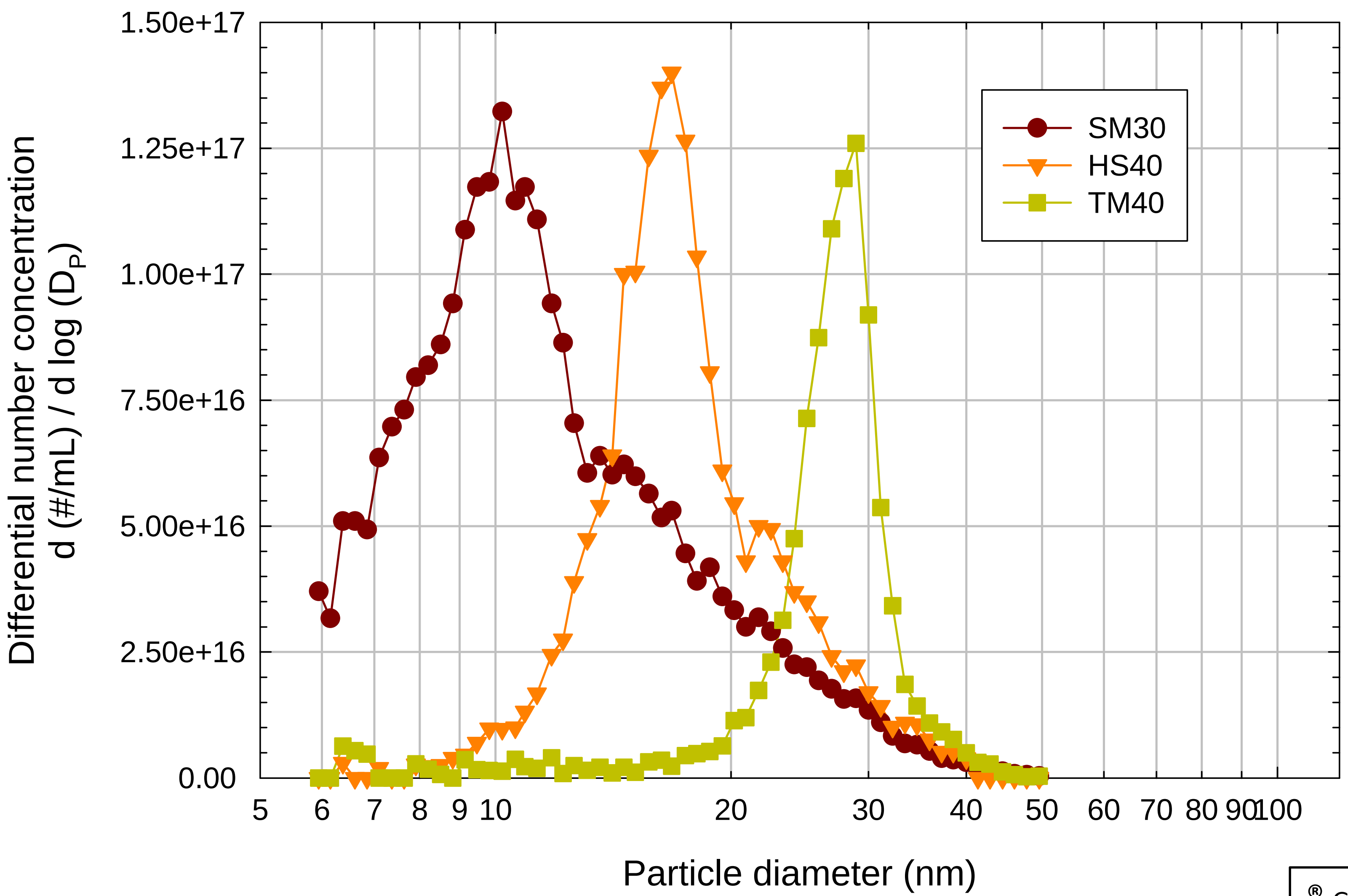




# Sizing of gold nanoparticles from BBI



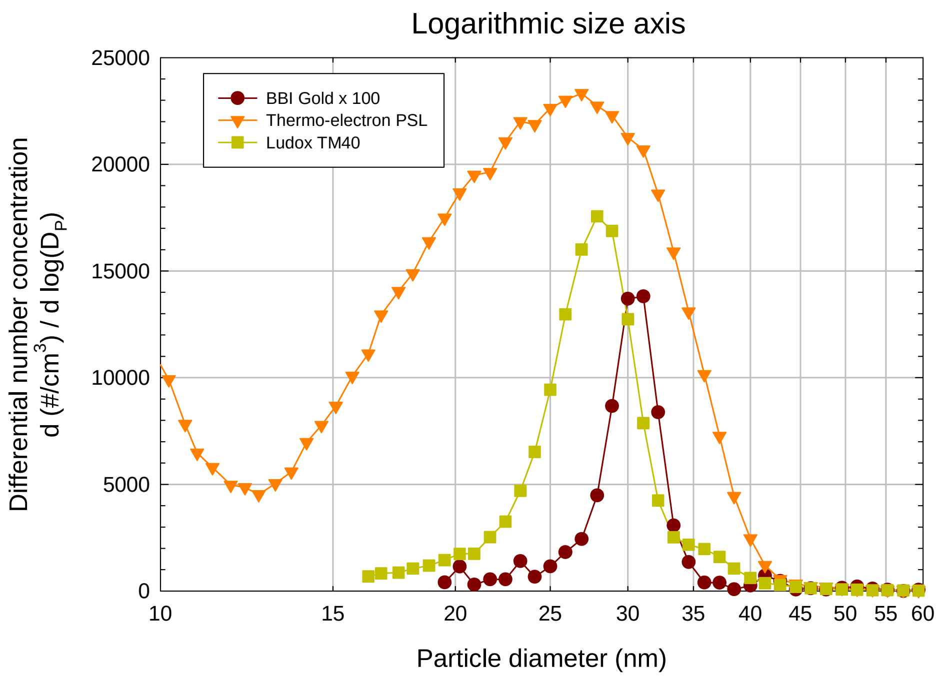



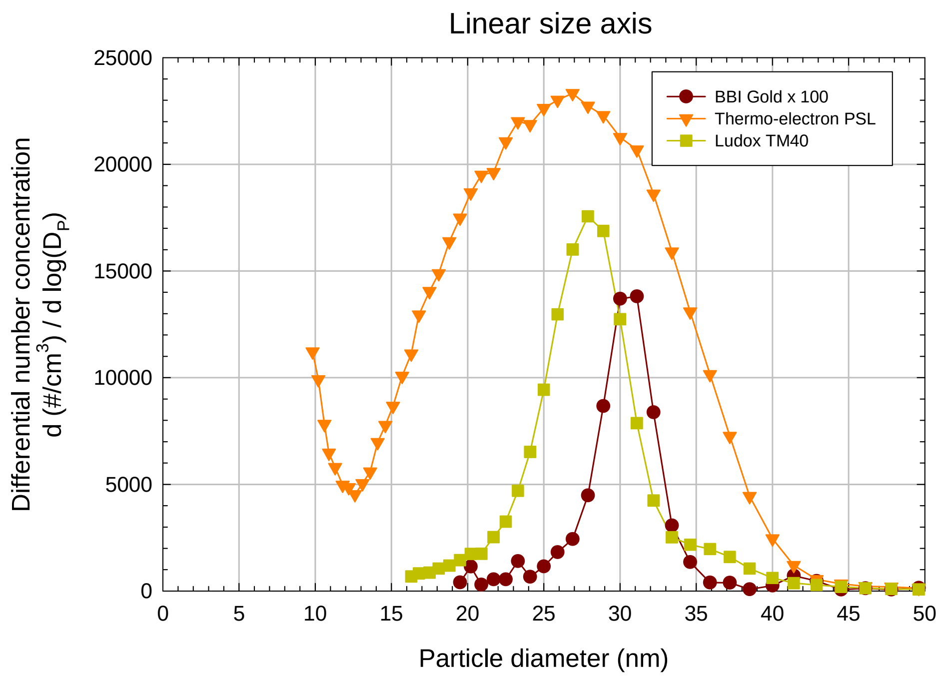
# Size distributions of Ludox® Colloidal Silica Particles




® Grace Davison

# PSDs of particles used in tests



# Filter-particle attraction

| | Hamaker Constant in water (1E-13 ergs) | | |
|---|---|---|---|
| Membrane | Gold | PSL | Silica |
| Polyethylene | 4.0 | 0.52 | 0.49 |
| Polyacrylic acid | 3.3 | 0.43 | 0.40 |
| PTFE | 0.12 | 0.016 | 0.015 |

Hamaker constants estimated using Lifshitz Theory.

# Anticipated particle capture mechanisms

- PSL
  - Particle zeta potential (-16 mV) predicts a moderate particle-membrane repulsive force in most cases.
  - Particle composition predicts a moderate 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 large 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 moderate particle-membrane attractive force in most cases.
  - Tests with multiple membrane types have indicated little if any non-sieving particle capture.

# Particle “real-worldliness”

- PSL
  - The particles are synthetic 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.

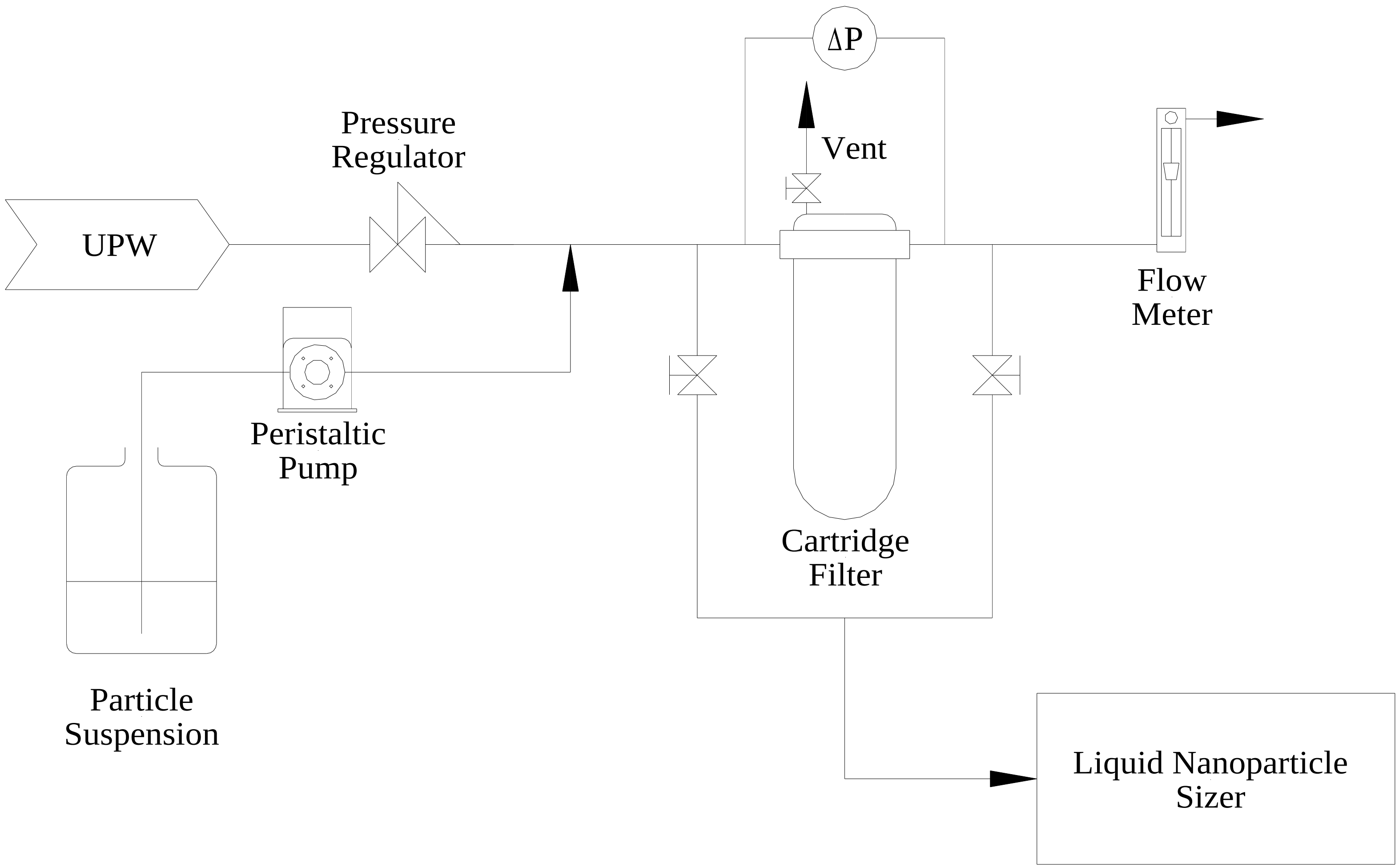
# Particle comparison

| 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 |

# Filter cartridge testing

- Cartridges were challenged with 3 types of nominally 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 following the initial test.

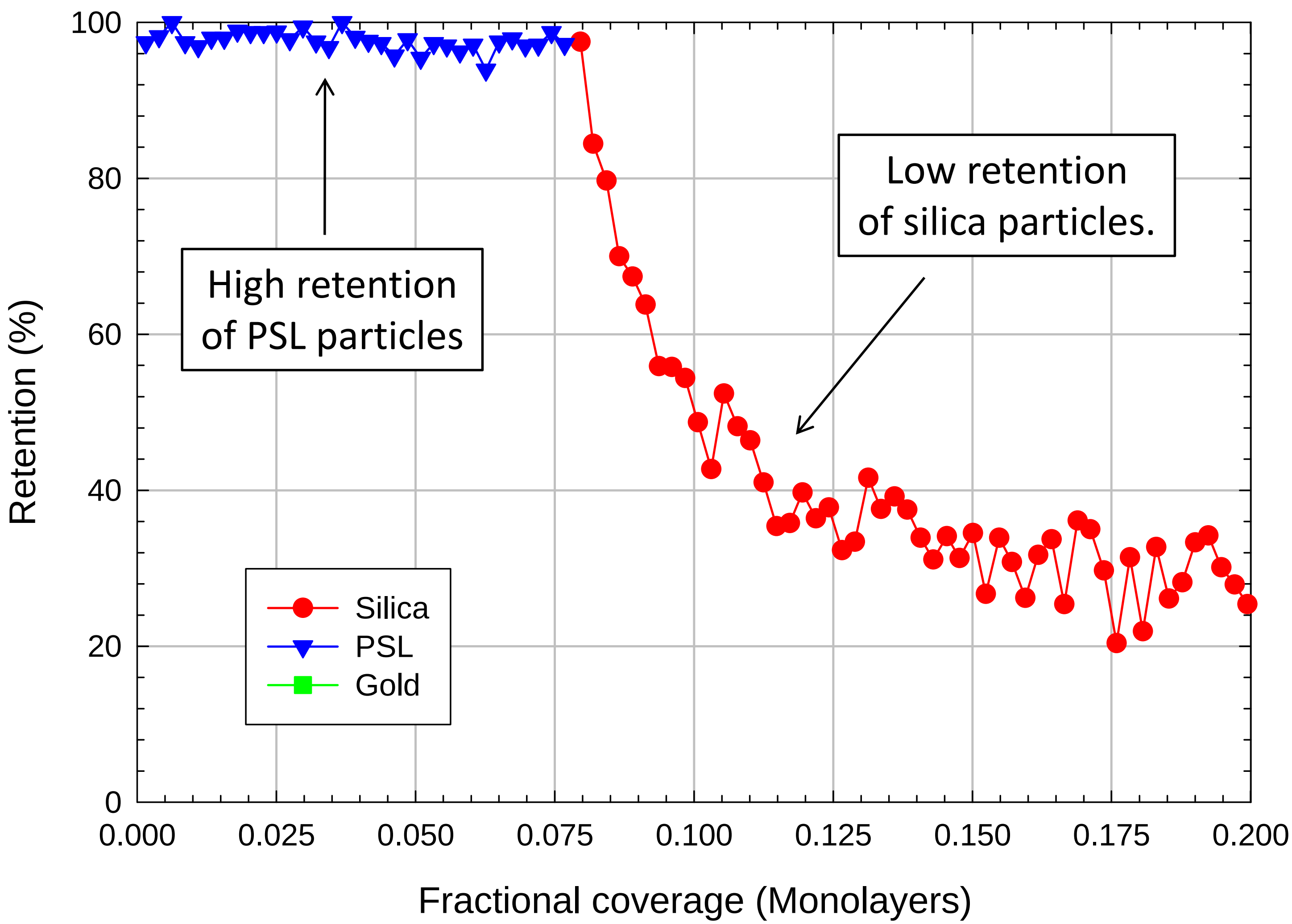
# Filter test system schematic



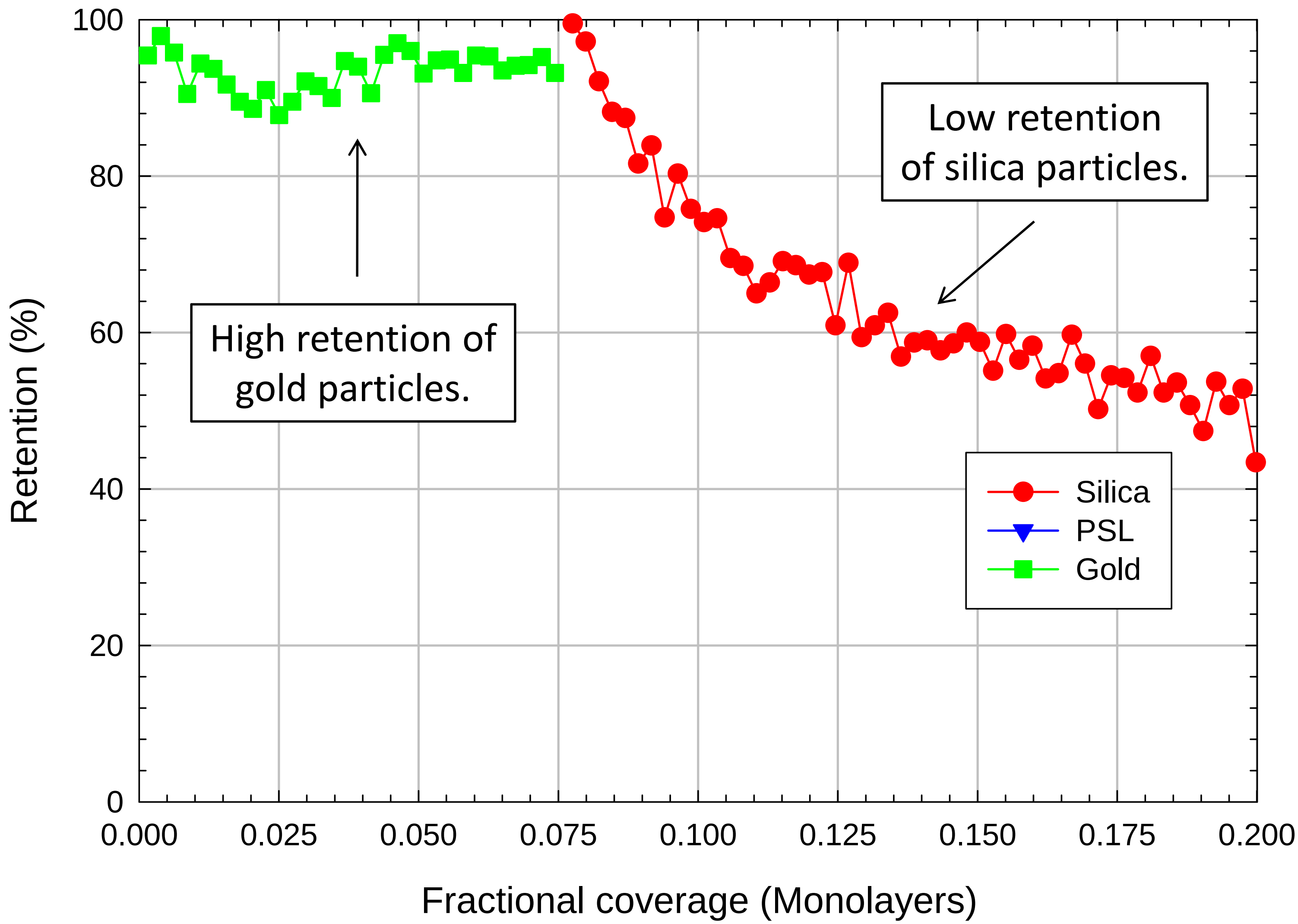
# Test Procedure

- The cartridges was flushed until the filtrate approached the system background concentration ($10^6$/mL > 10 nm).
- The filter was challenged with $2x10^8$ particles/mL (~6ppb).
- The challenge concentration was verified.
- The face velocity  throughout the test was 0.11 cm/min.

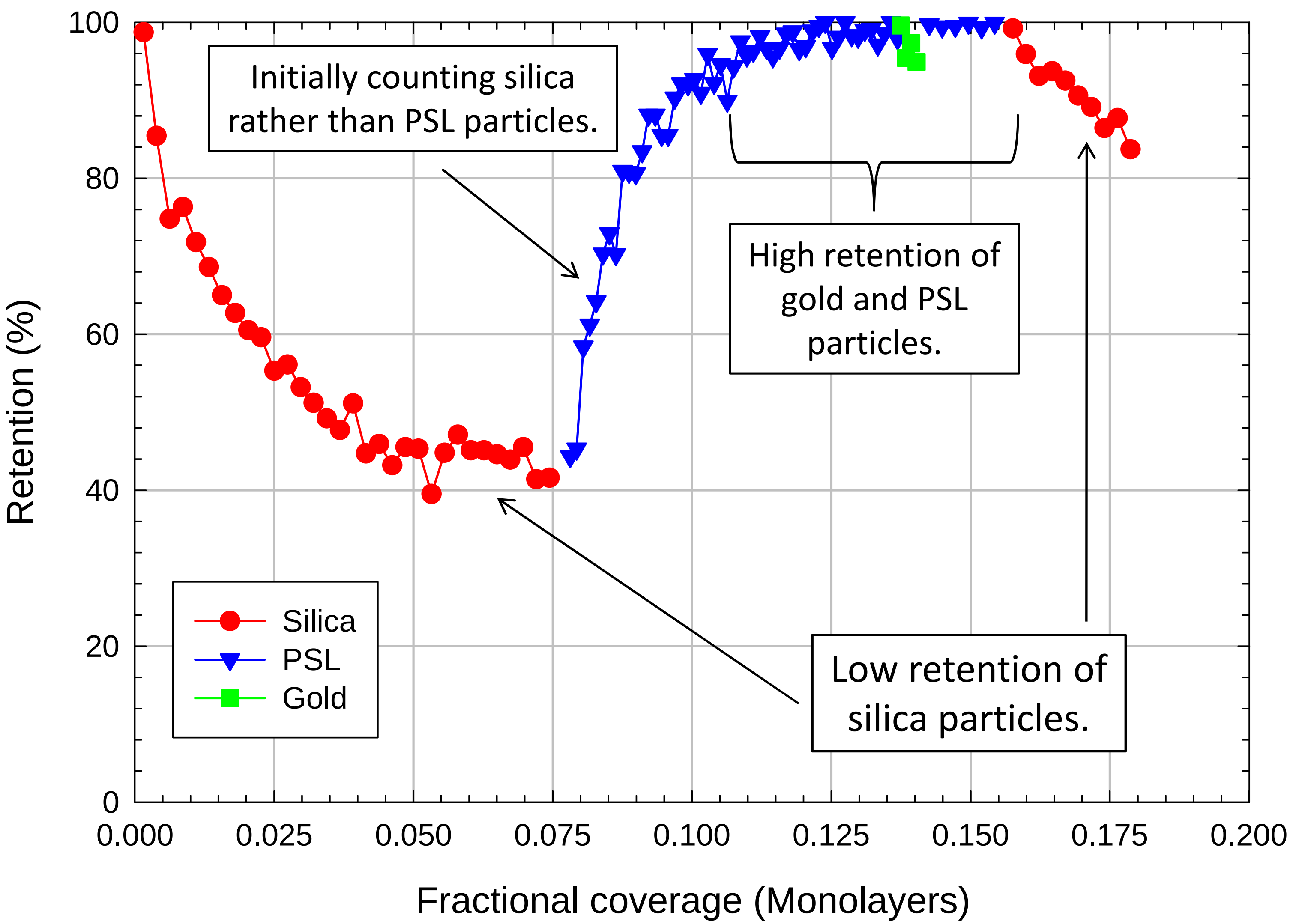
# Retention of PSL followed by silica
## (Cartridge #1)

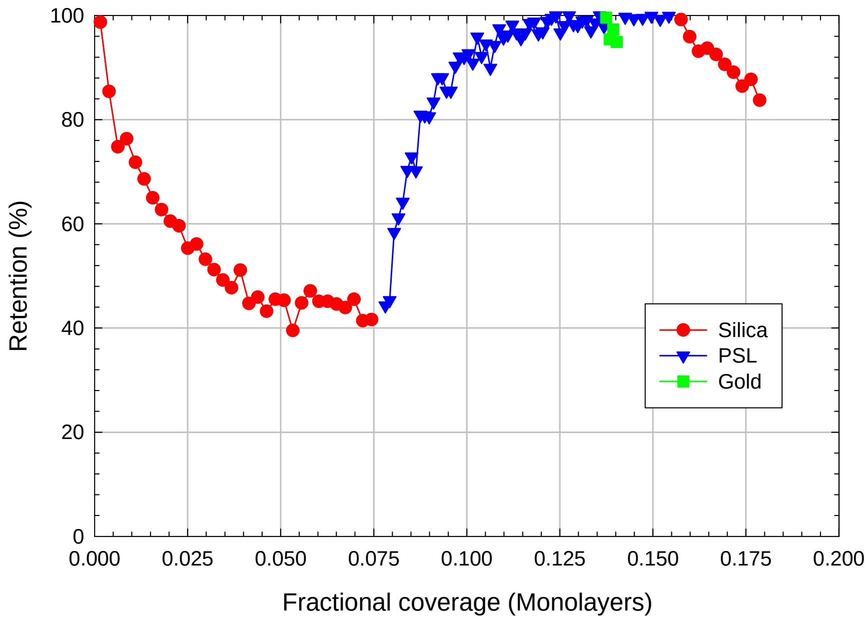


# Retention of gold followed by silica
## (Cartridge #2)



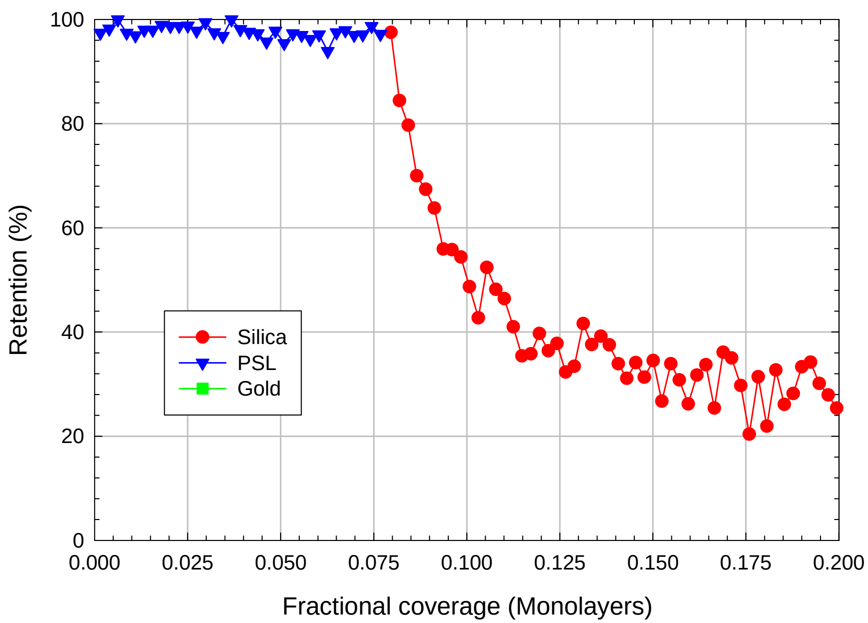
# Retention of multiple particle types
(Cartridge #3)

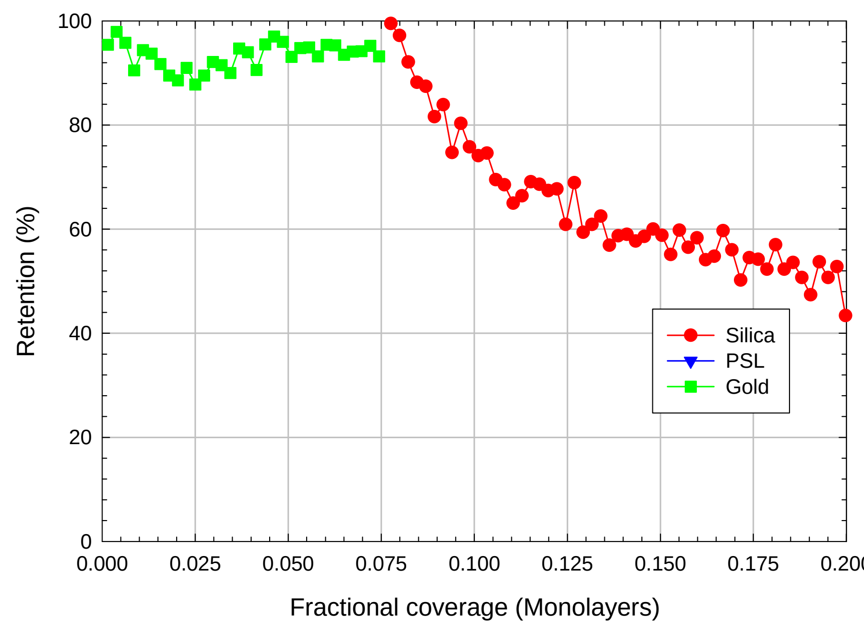





# Retention of different 30nm particles types by a commercially available UPW filter cartridges

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



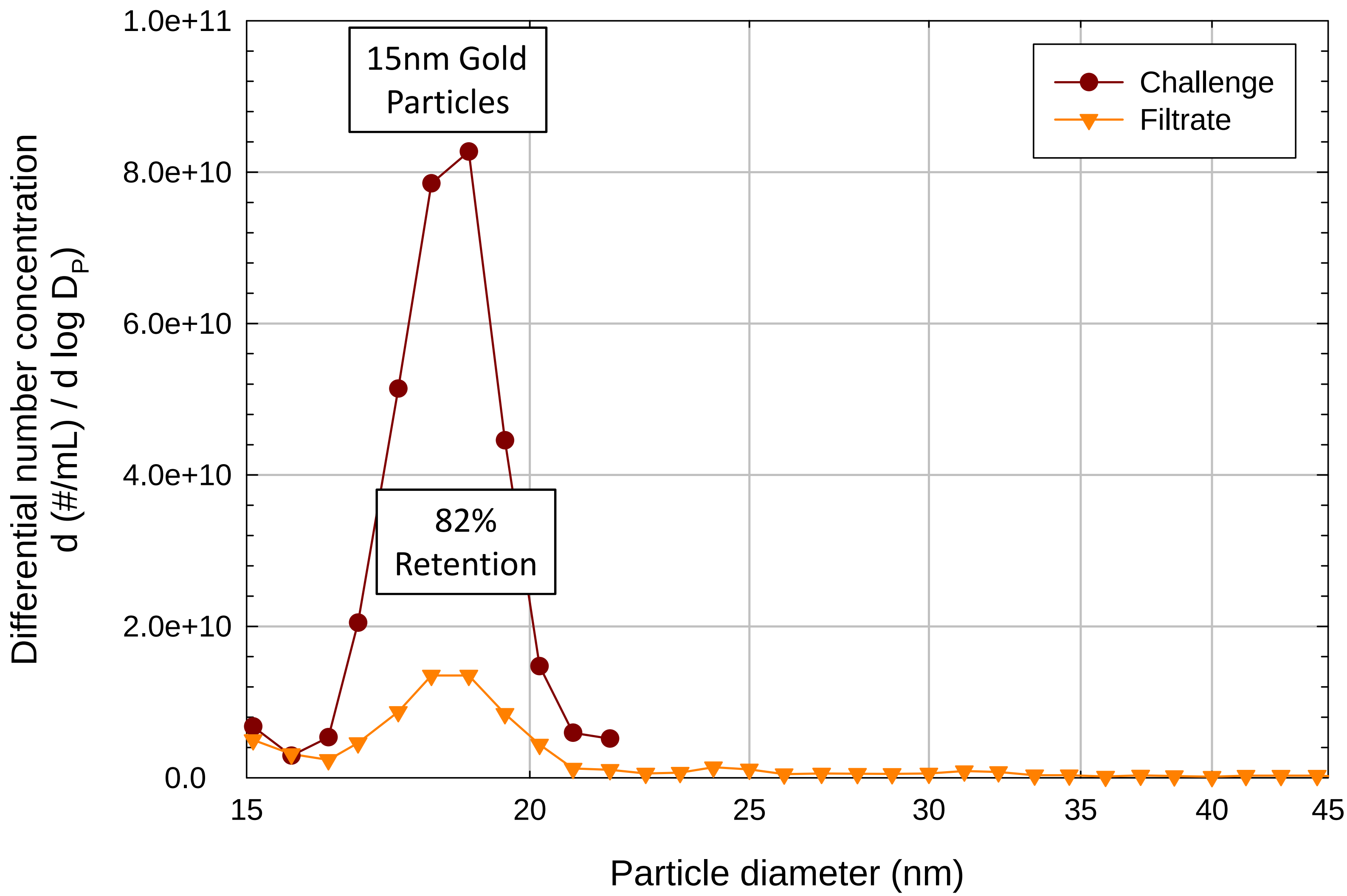



# Retention of a mixture of 15 nm gold and 30 nm silica by a commercially available UPW filter

- A cartridge was challenged with $3x10^8$/mL 15 nm gold particles for 3 hours.
- This was followed by a 3 hours challenge with a mixture of $3x10^8$/mL gold + $3x10^8$/mL silica particles

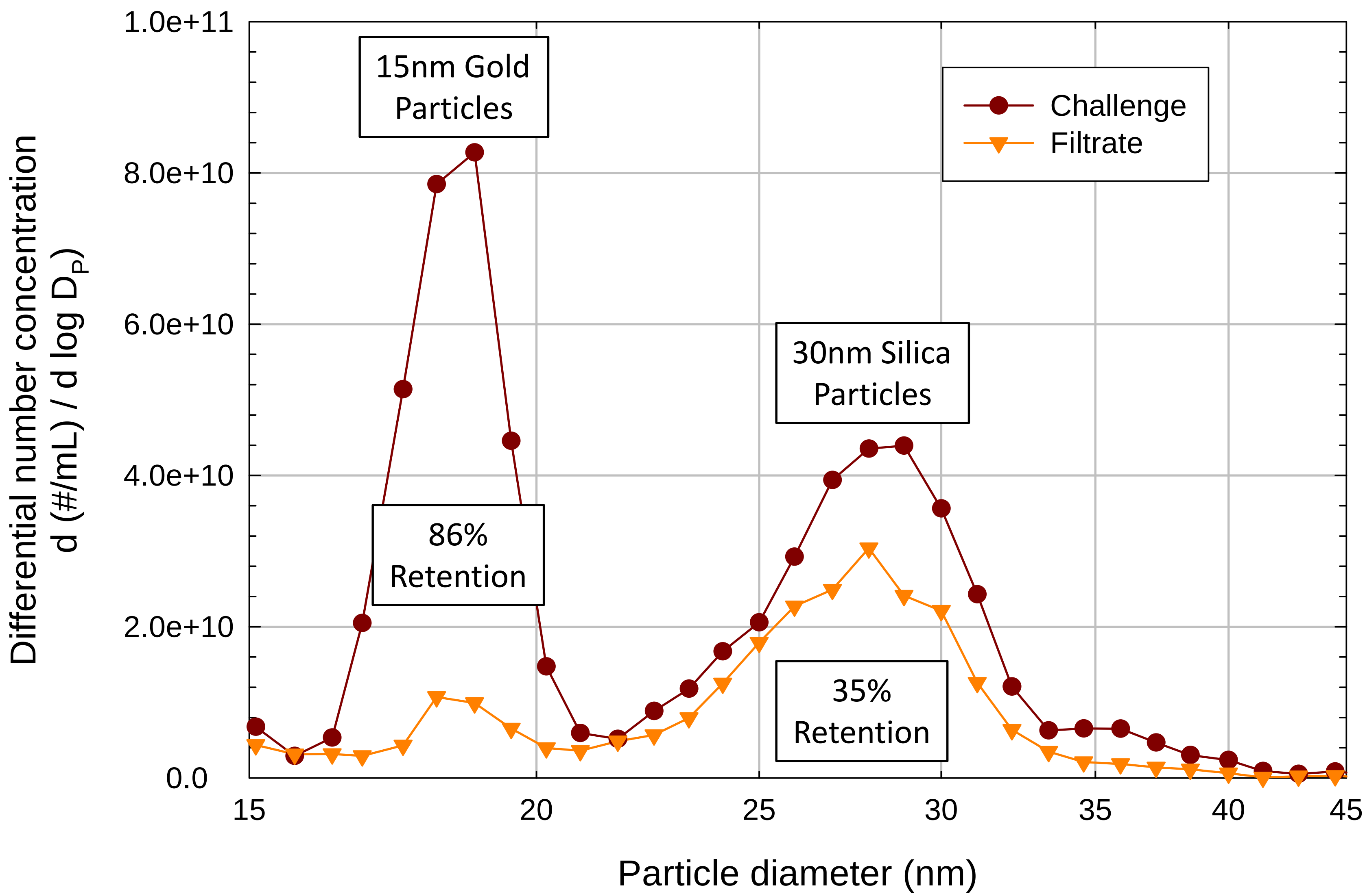
# Retention of gold particles

## (Concentrations at 3 hours)



# Retention of a mixture of particle types

## (Concentrations at 6 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 removed by multiple mechanisms.
- Silica is the recommended particle type for UPW filter retention testing.

# Summary and conclusions

- Test methods to measure the retention of sub-30nm 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
- The chemical compositions of the filter and particle determine what particle capture mechanisms are active.
- Filter retention tests performed with PSL, gold, and silica 30nm particles indicate that capture of silica particles is predominately by sieving while other, more efficient, mechanisms are active in capturing PSL and gold particles.
- Silica is the recommended particle type for UPW filter retention testing.