Measurement of 28 nm particle removal from liquids by high purity filters

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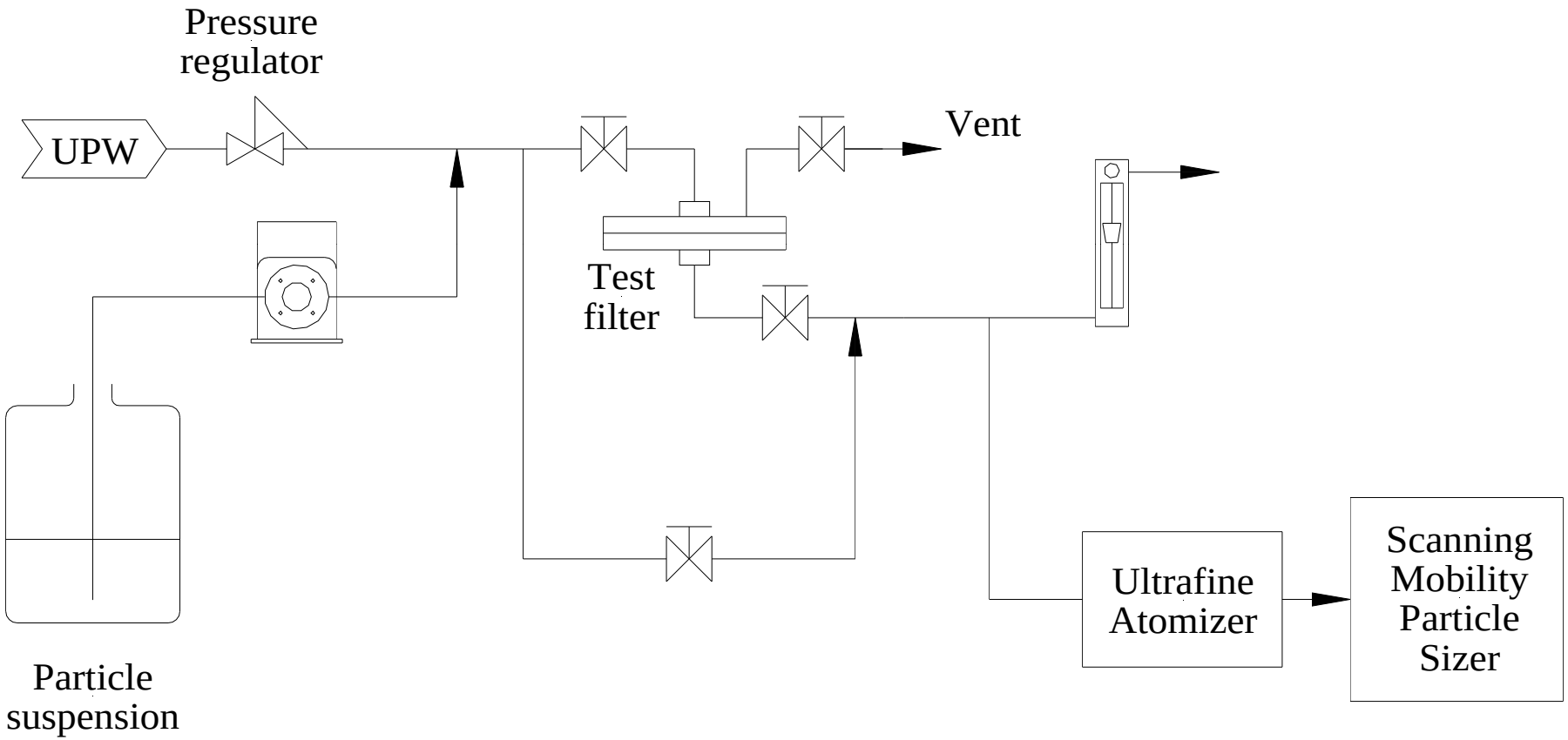
Abstract

The critical features size of state-of-the-art semiconductor devices is on the order of 50 nm and expected to decrease to ~ 20 nm by 2015. Particles on the order of ½ the feature size in process liquids used during device manufacturing can reduce device manufacturing yield and finished device reliability. Microfilters and ultrafilters with pore size ratings below 50 nm are often used to control particle concentrations in these liquids. However, the ability of the filters to remove particles is typically measured using optical particle counters with a minimum detection limit of 50 nm or larger.

This paper describes a new technique that allows measurement of removal of particles as small as 10 nm in diameter from liquids. In this technique filters are challenged with particles ranging from 10 to 100 nm in diameter. Total particle concentrations ranging from 10^7/mL to 10^{10}/mL $\geq$ 10nm are used. Filter inlet and outlet concentrations are measured using ultrafine atomization/scanning mobility particle sizing (UFA/SMPS), a recently developed technique. In this technique a very fine mist of particle-laden water is created, the water in the mist droplets is evaporated and sizes and numbers of the remaining particles are measuring using a scanning mobility particle sizer. This technique allows very accurate resolution of particle size with 64 size channels between 10 and 100 nm.

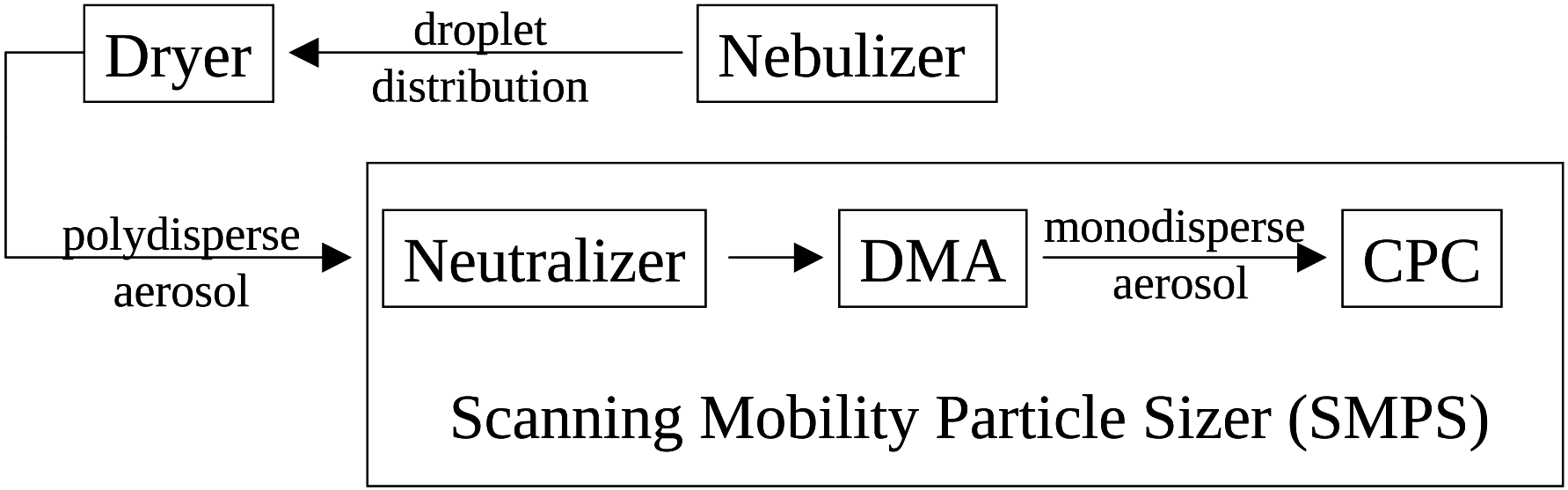
Examples of retention of poly-dispersed polystyrene latex (PSL) and mono-dispersed 28 nm colloidal silica particles by filters with pore size ratings between 20 and 100 nm are shown. The effect of particle loading on retention by these filters is examined.

Test system schematic

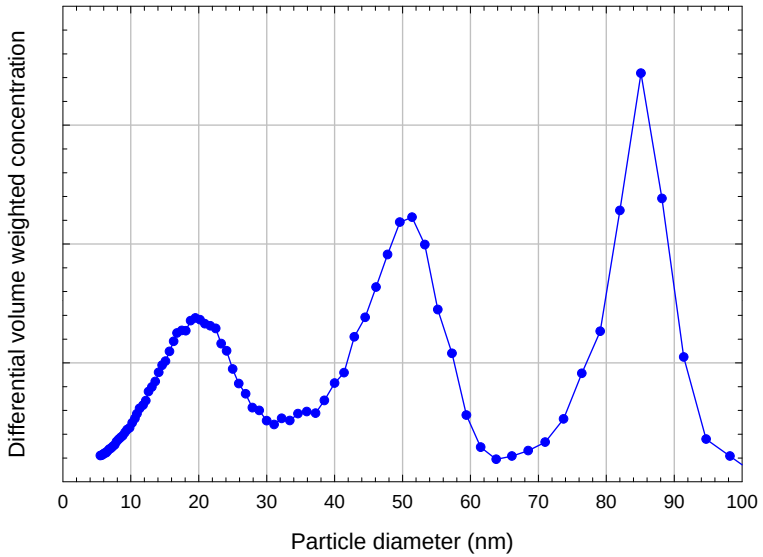


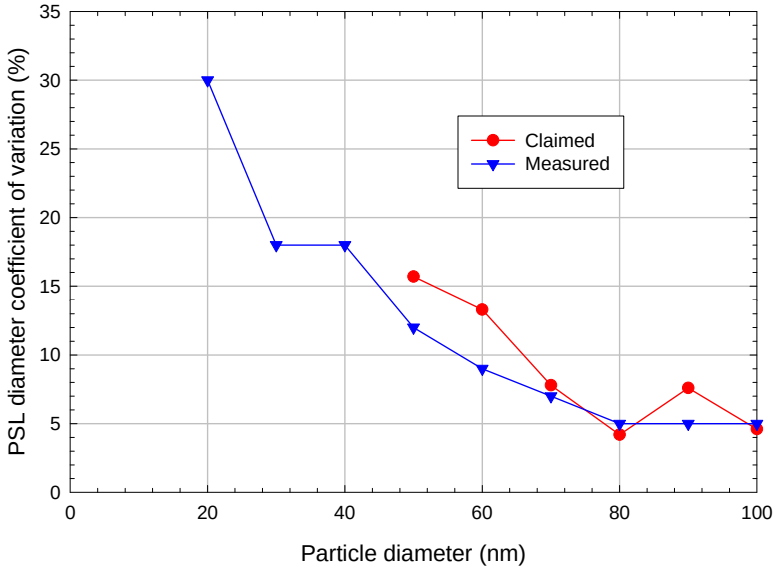
Particle Measurement Technique

Ultrafine atomization/scanning mobility particle sizing



Measurement technique
sizing accuracy and resolution





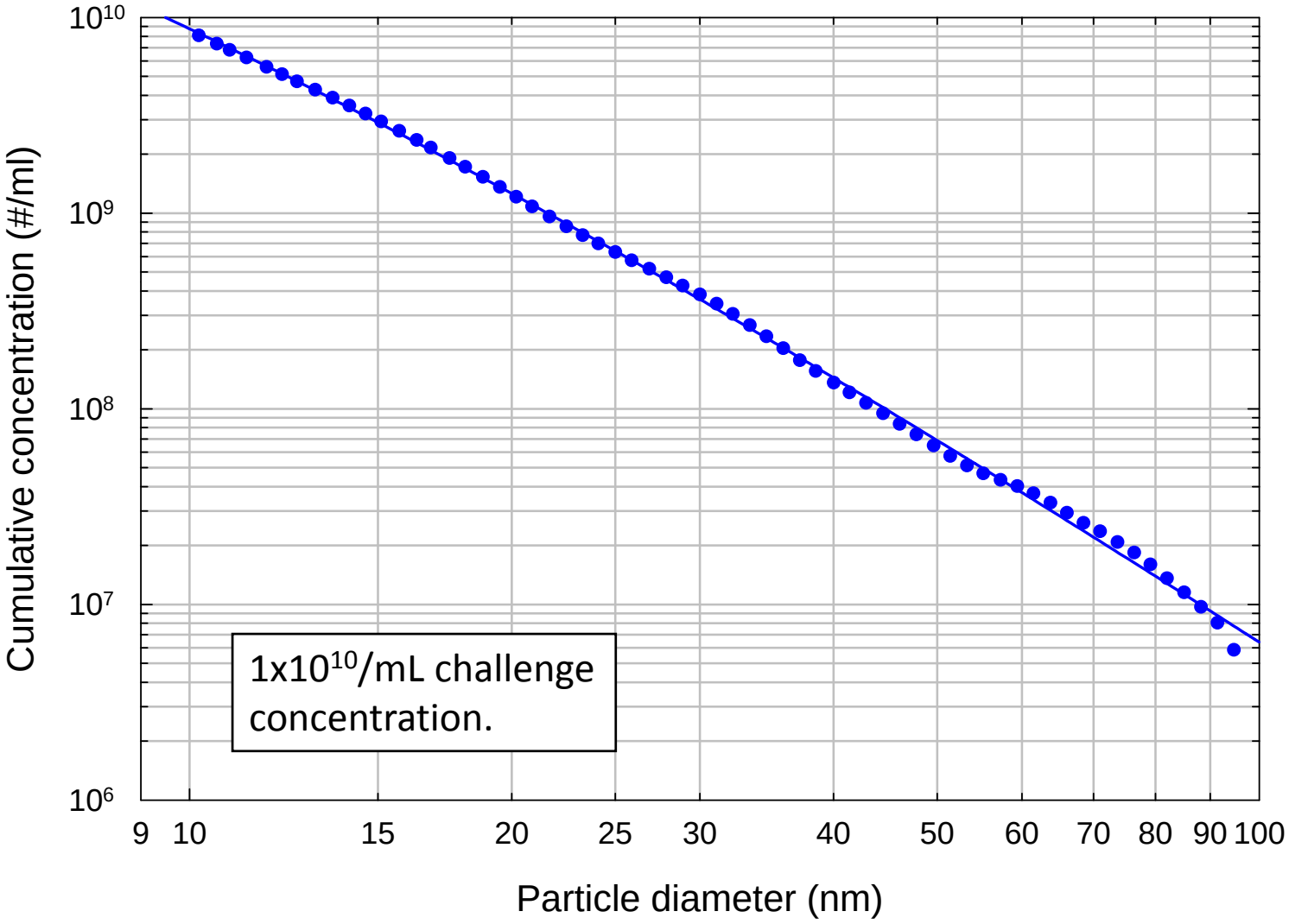
- Simultaneous measurement of 20, 50, and 80 nm polystyrene latex (PSL) spheres.
- Instrument can easily resolve the 3 PSL size sizes.

- Measured and supplier claimed PSL size variations are similar indicating no discernible variation due to instrument.

Filter capture of polystyrene latex particles

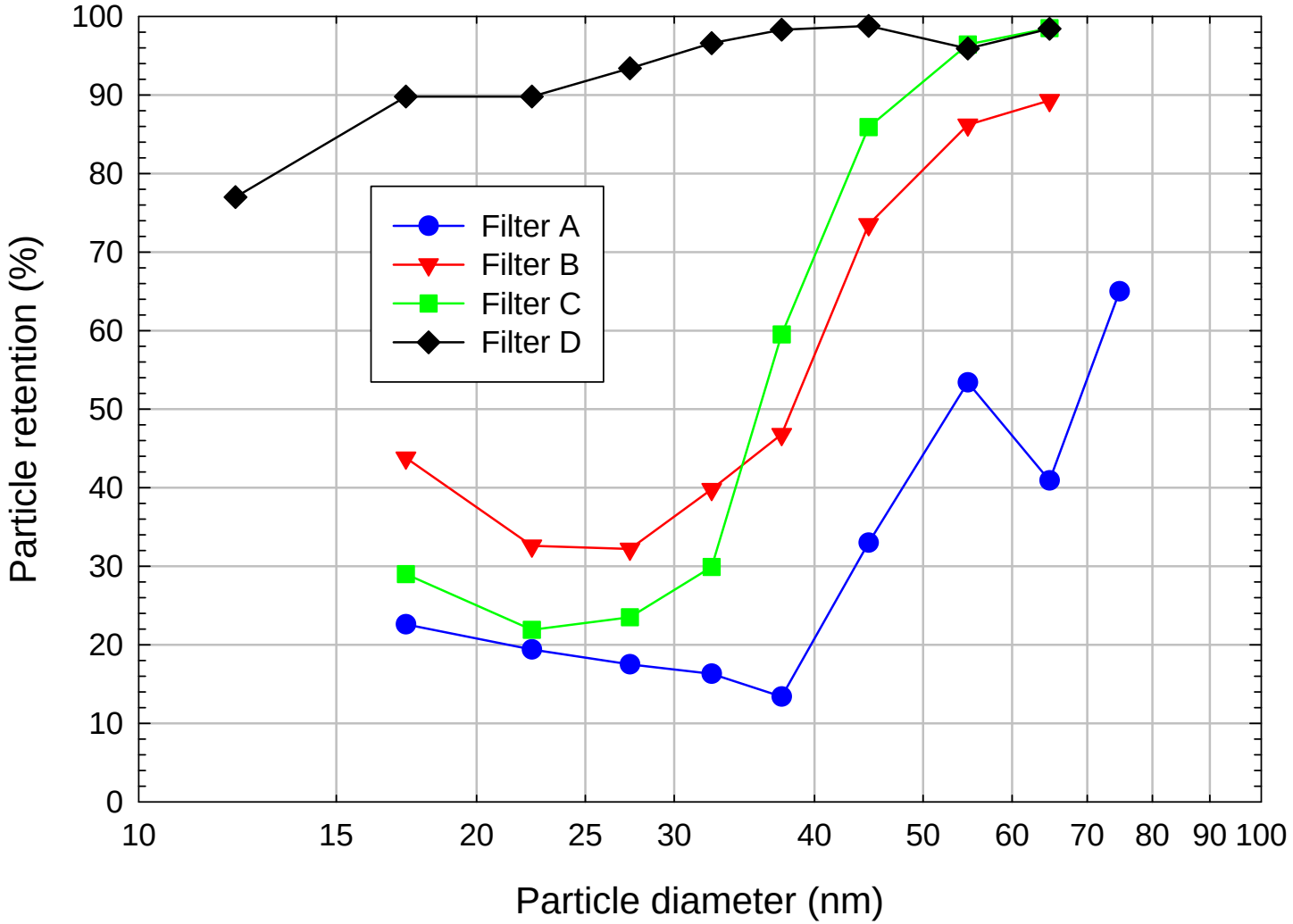
- Filters were challenged with a mixture of nine sizes of polystyrene latex (PSL) beads with median diameters ranging from 20 to 100 nm.
- Prior to the particle challenge the filters were flushed with ultrapure water (UPW) to eliminate particle shedding.
- Four challenge particle concentrations were used ranging from $3x10^{8}$/mL to $1x10^{10}$/mL $\geq$ 20nm (6 to 200 µg/lit).
- Particle concentrations upstream and downstream of the filter were measured using ultrafine atomization/scanning mobility particle sizing (UFA/SMPS).
- Filter particle retention was measured as a function of particle size and loading.

PSL challenge particle size distribution



Filter retention of poly-dispersed PSL particles

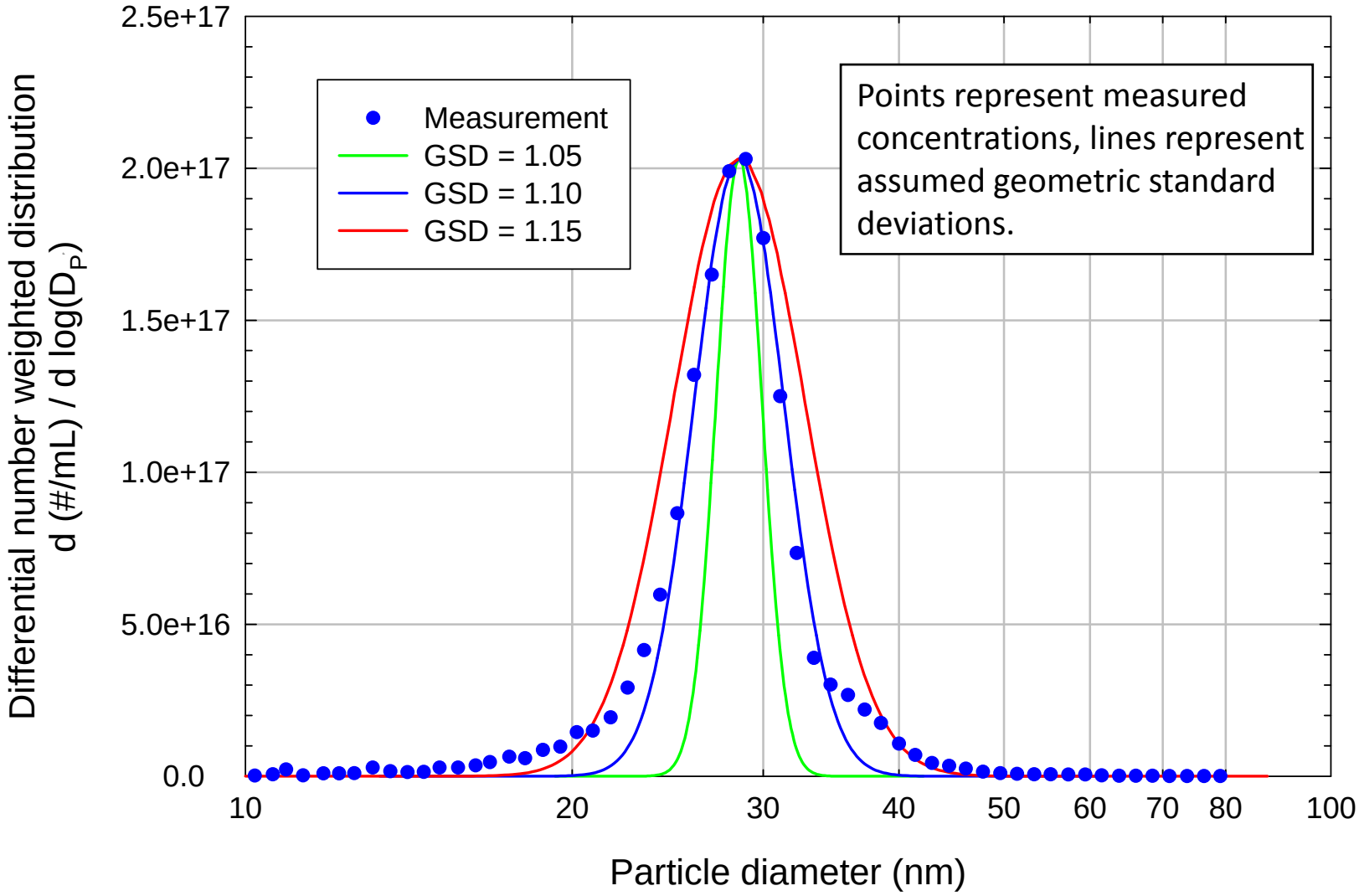
(3x10^9/mL; 1-3 monolayer coverage)



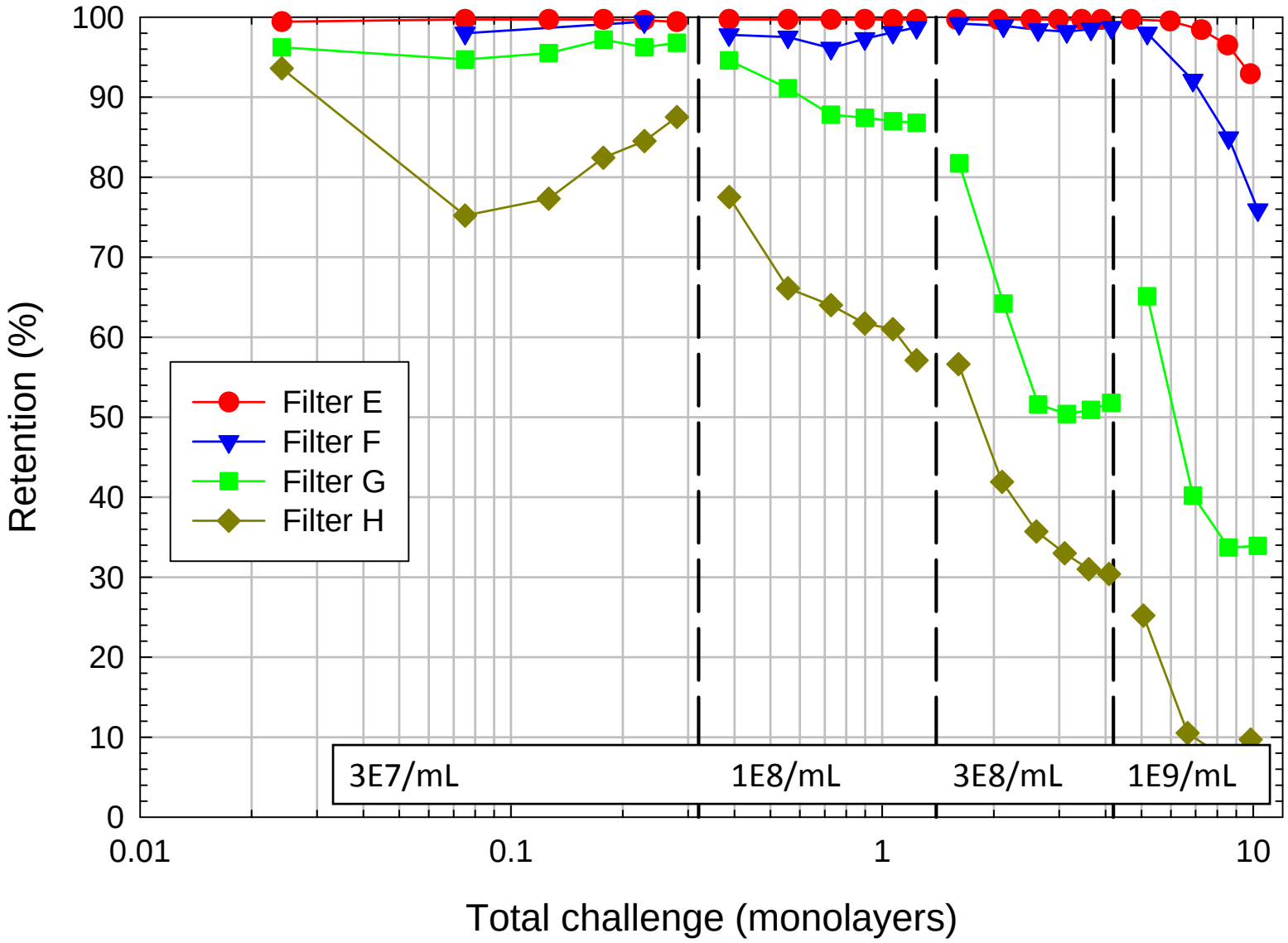
Filter capture of colloidal silica particles

- Filters were challenged with mono-dispersed silica particles with a median diameter of 28 nm.
- Prior to the particle challenge the filters were flushed with UPW to eliminate particle shedding.
- Four challenge particle concentrations were used ranging from $3x10^7$/mL to $1x10^9$/mL (0.7 to 23 µg/lit).
- Particle concentrations upstream and downstream of the filter were measured using ultrafine atomization/scanning mobility particle sizing (UFA/SMPS).
- Filter particle retention was measured as a function of particle size and loading.

Silica challenge particle size distribution



Retention of 28nm Silica particles by 4 filter types



Summary

- Test methodology to measure retention of particles as small as 10 nm by membrane filters has been developed.
- Particle concentrations are measured using ultrafine atomization/scanning mobility particle sizing (UFA/SMPS).
- The method showed clear differences between retentions of both poly-dispersed polystyrene latex particles and mono-dispersed 28 nm silica particles by different filter types.