

Particle Size and Concentration Standards for Sub-100 nm Applications

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Introduction

- Calibration of liquid optical particle counters ideally requires a polystyrene latex (PSL) standard with a known particle size distribution (particle diameter vs **concentration**).
- Measuring the PSD of colloidal suspensions below 200 nm has been difficult and has limited the availability of standards below this size.
- Using a newly developed liquid nanoparticle sizing system, PSD of PSL suspensions can be measured down to 5 nm allowing preparation of concentration standards below 100 nm.
- The testing of liquid filter retention can also benefit from a full understanding of the particle size distribution (PSD) of the challenge solution.



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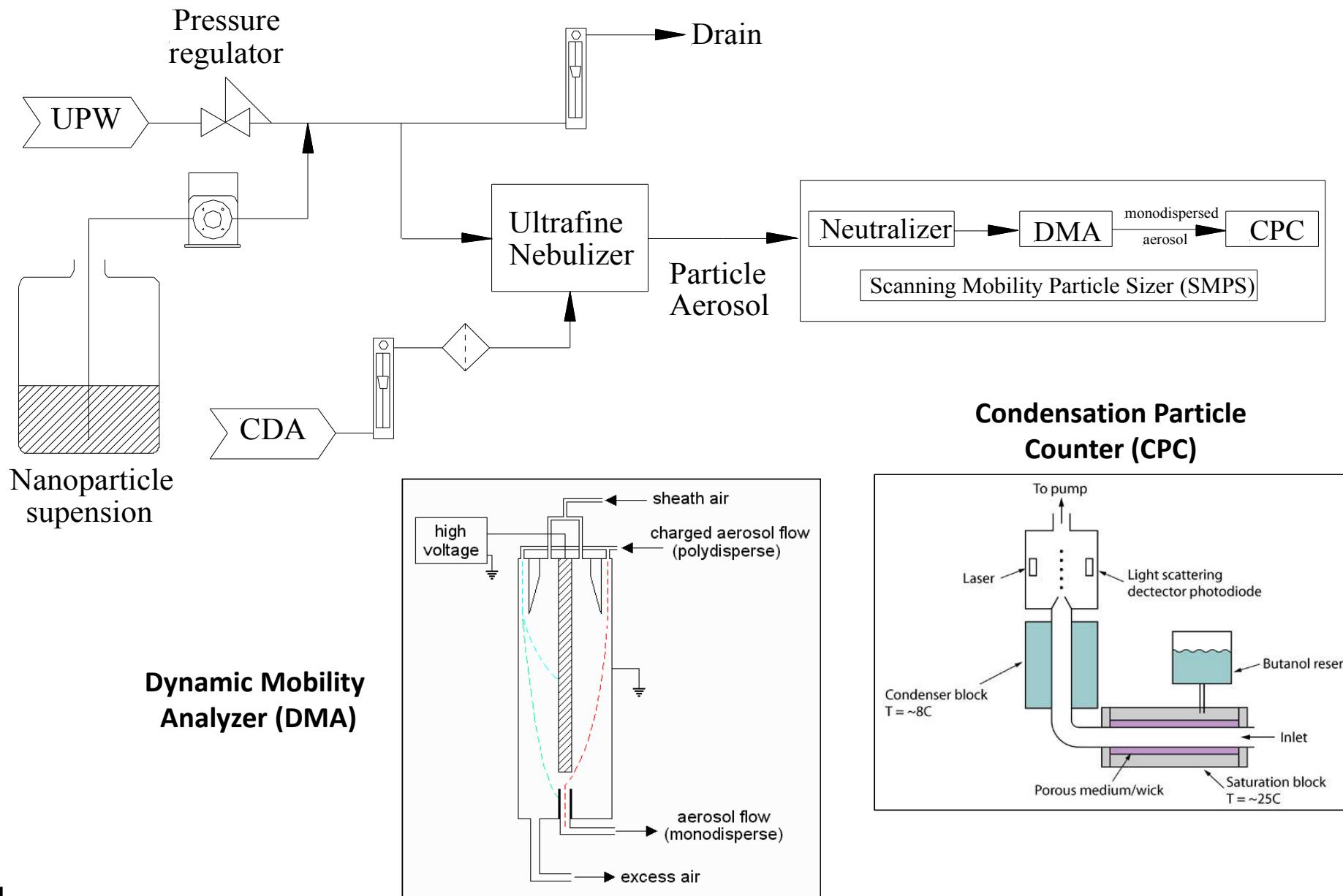
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Schematic of the Liquid Nanoparticle Sizing System



Liquid nanoparticle sizing system capabilities and benefits

- Broad operational range:
 - Single-particle quantitation from samples with particle concentrations at $2.0 \times 10^8 - 1.0 \times 10^{15}$ particles/mL.
 - Sizing range from 2.5 to 450 nm with 64 channels per decade resolution.
- 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.
- Aerosol instrument (DMA) is used by NIST to size PSL reference materials.



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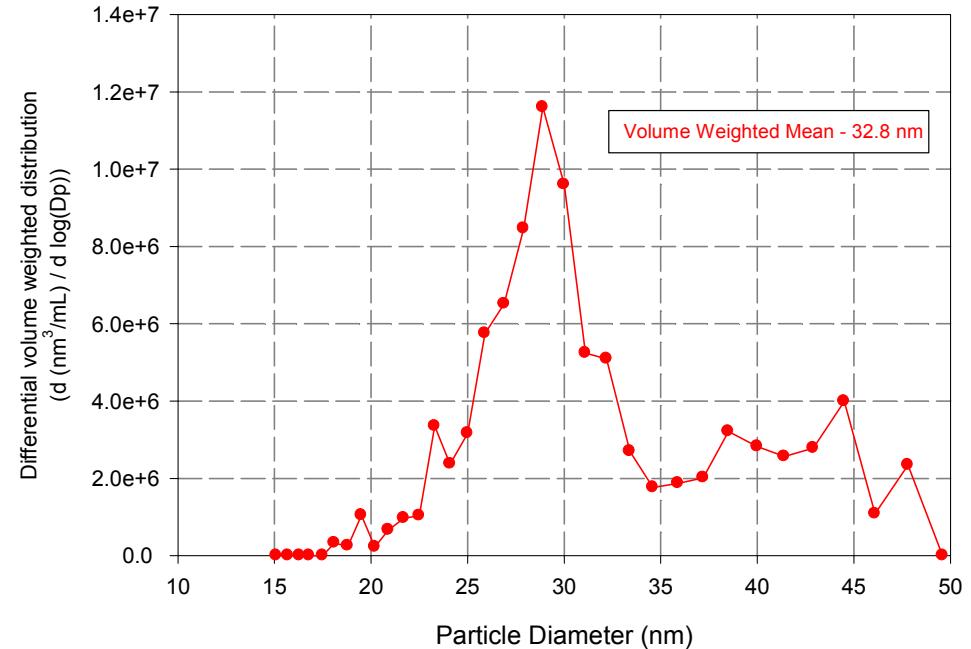
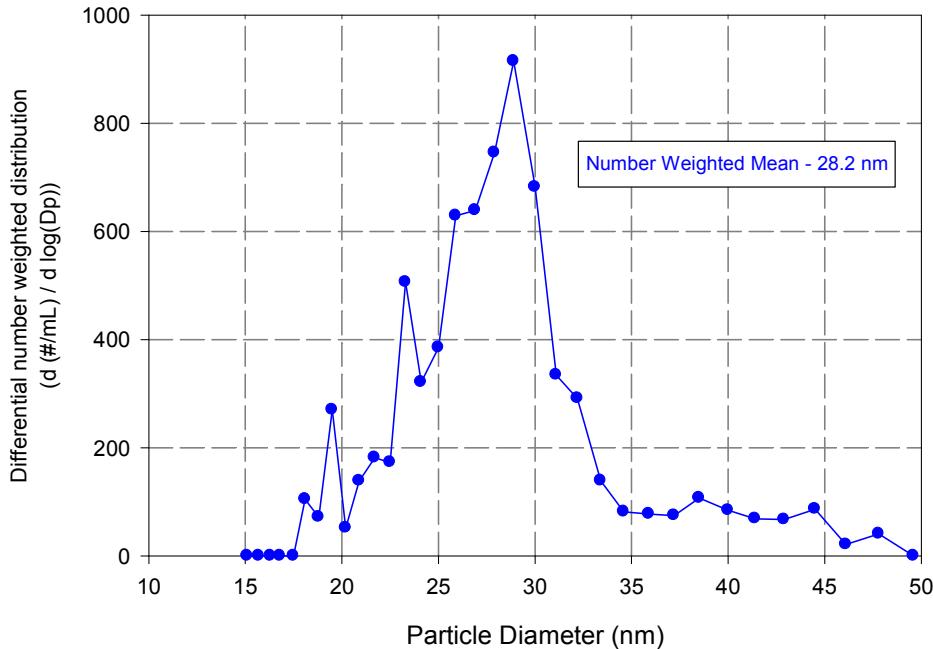
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NIST 30 nm gold analysis via liquid nanoparticle sizing



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

| Technique | Analyte Form | Mean Particle Size (nm) | Expanded Uncertainty |
|---|-----------------------------|-------------------------|----------------------|
| 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 |



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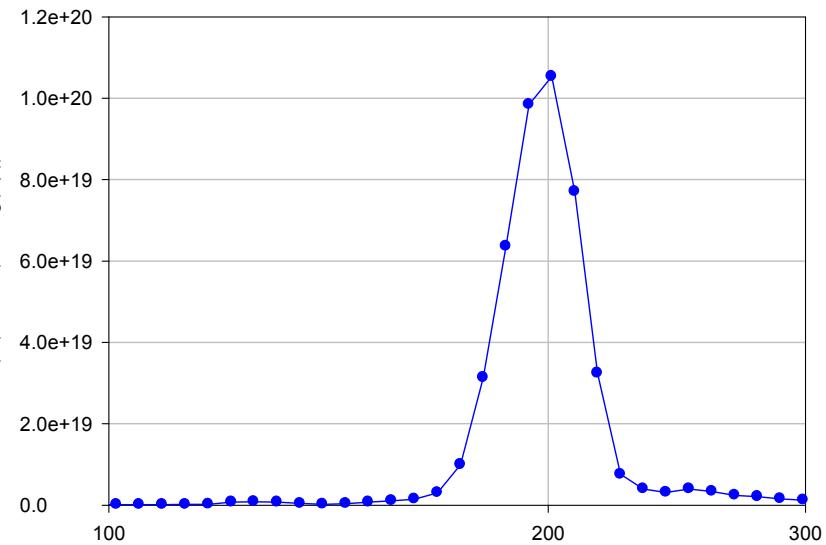
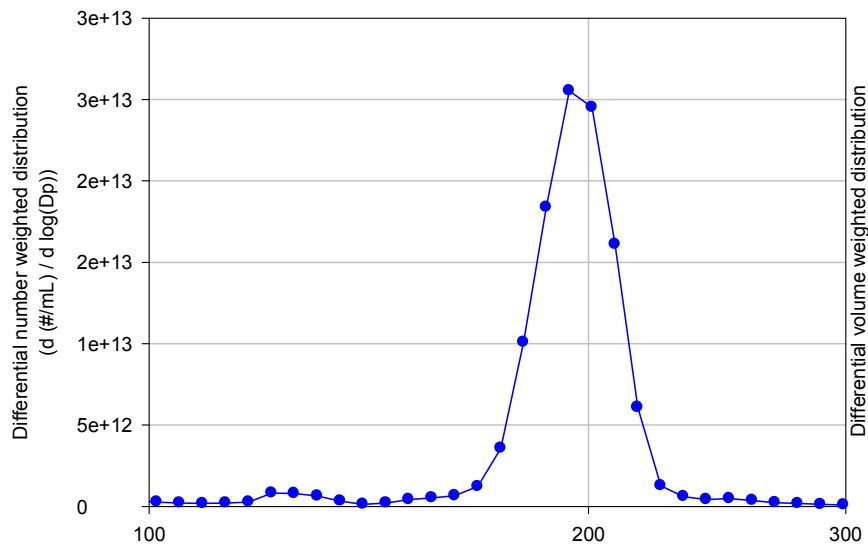
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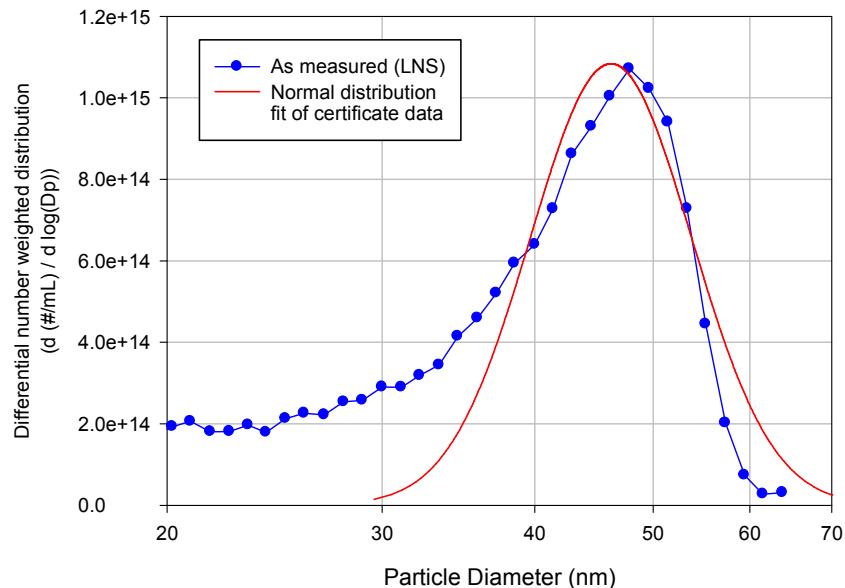
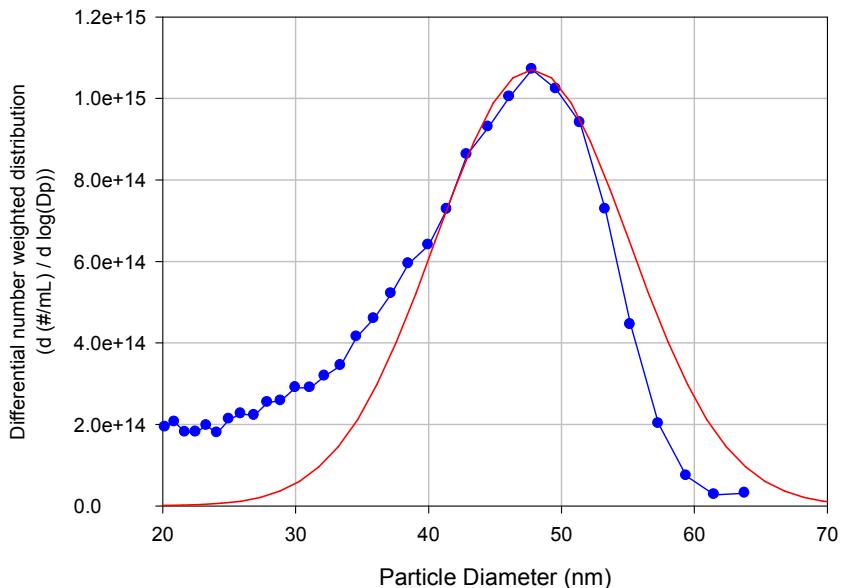
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Particle size distribution of polystyrene latex

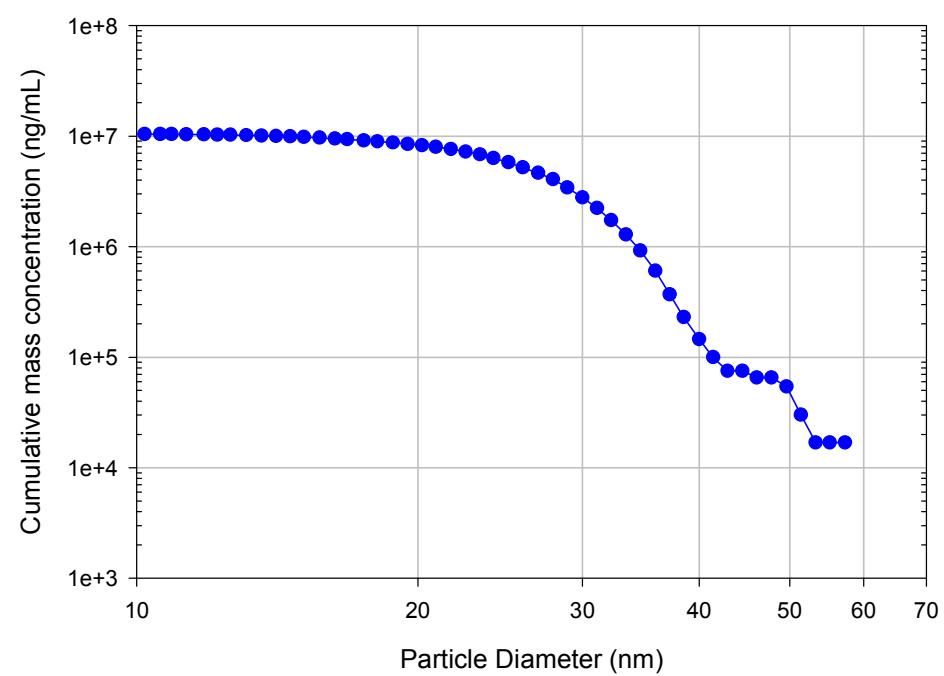
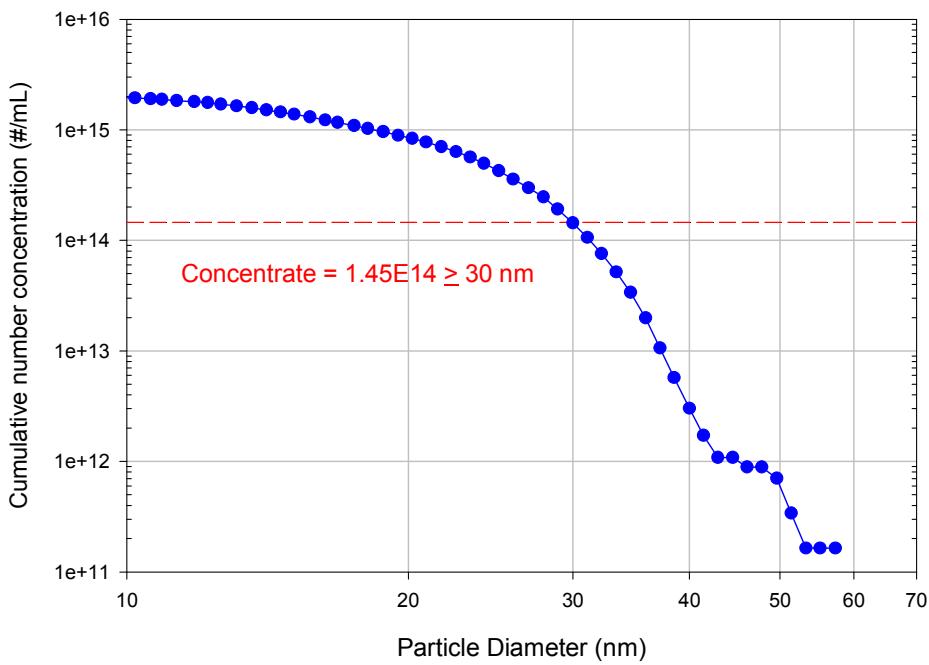
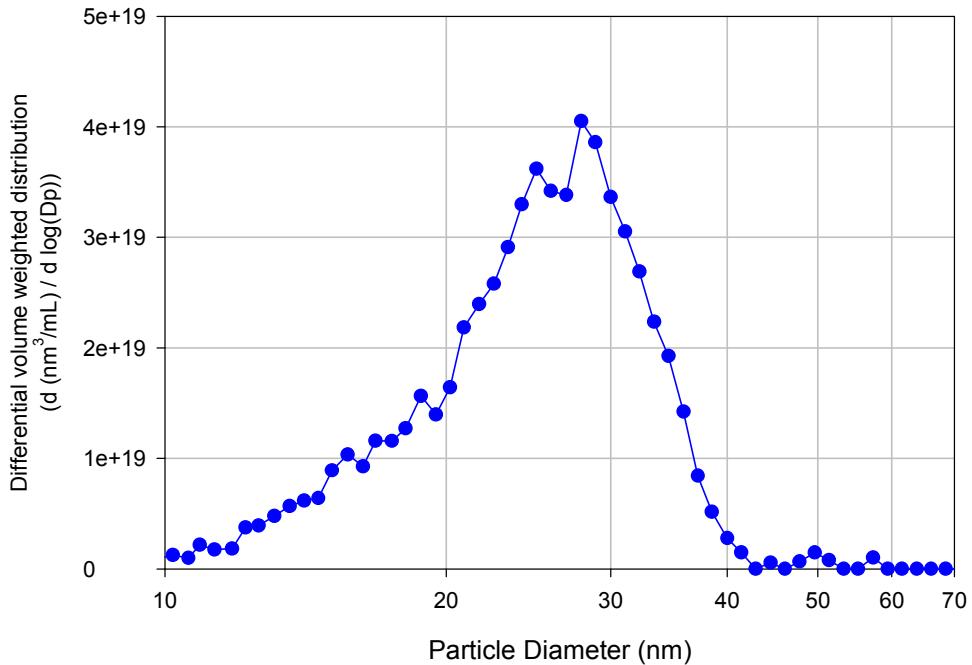
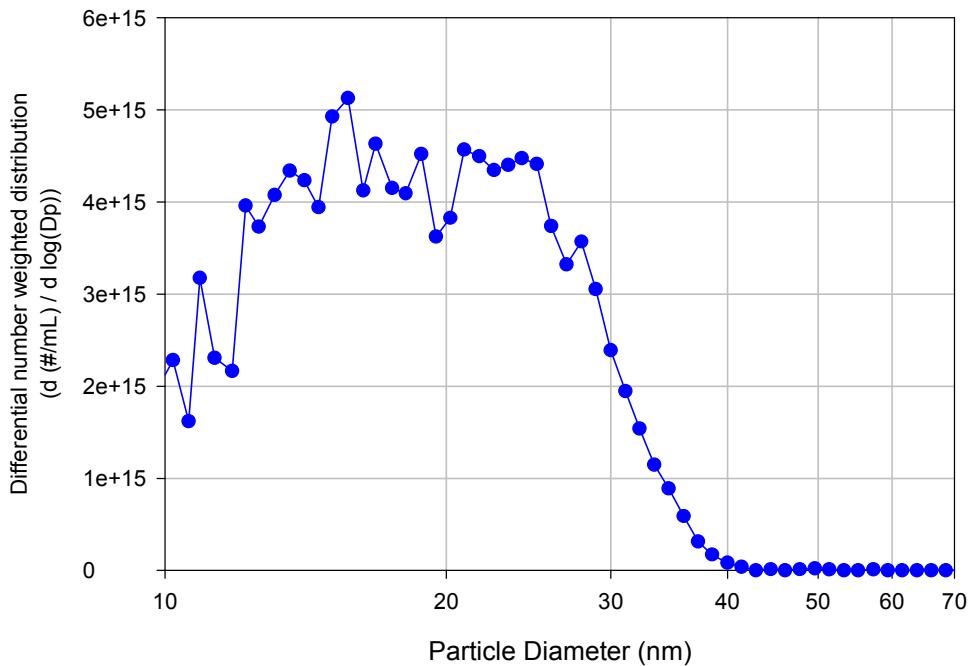
NIST Traceable 200 nm PSL
(Reported Size - 203 ± 5 nm)



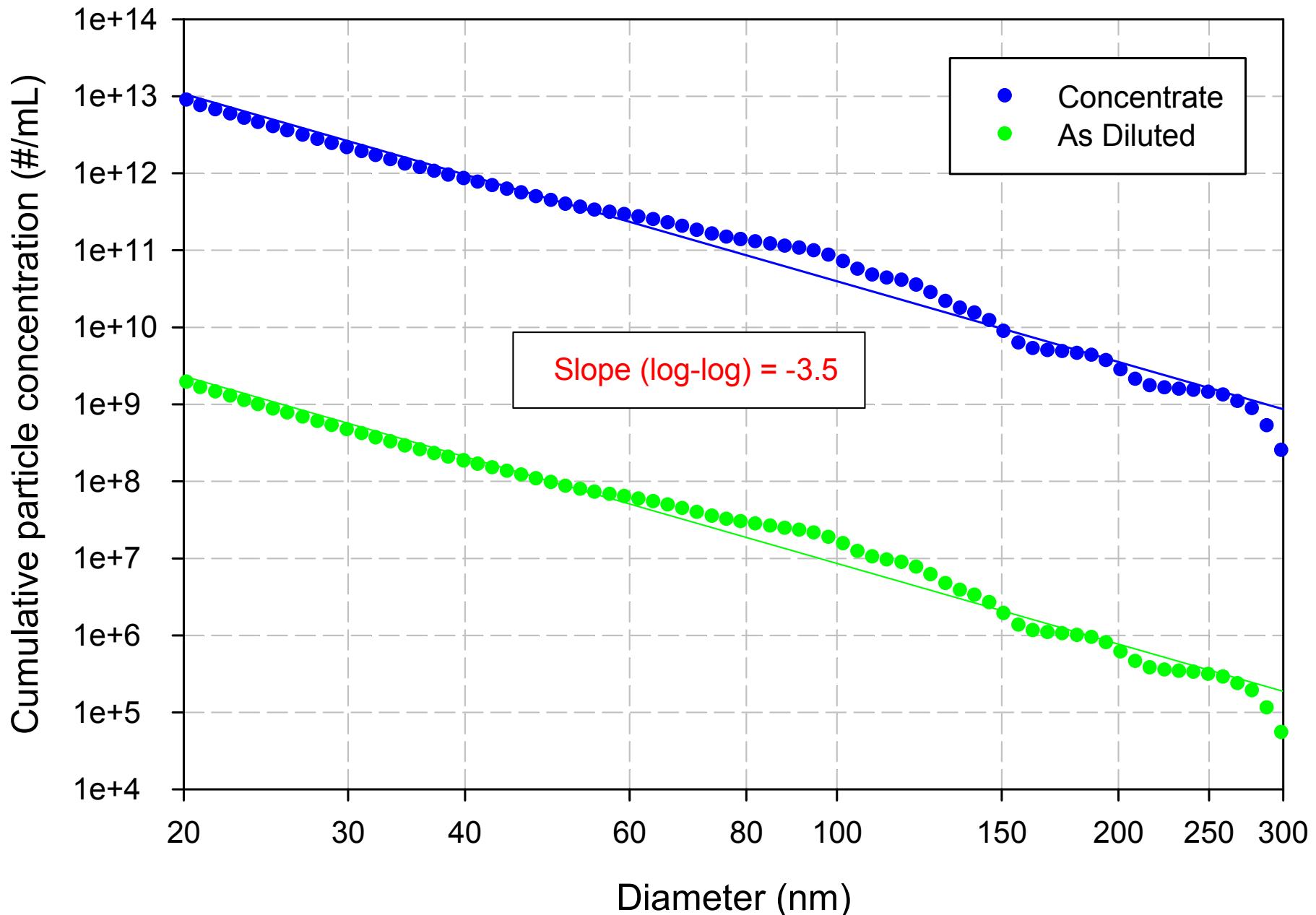
NIST Traceable 50 nm PSL
(Reported Size - 46 ± 2 nm)



NIST Traceable 30 nm PSL
(Reported Size - 30 ± 1 nm)

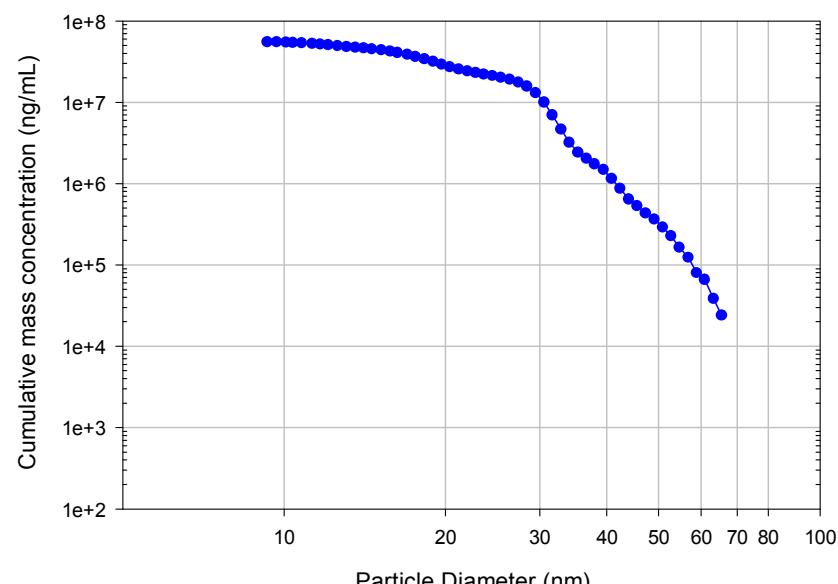
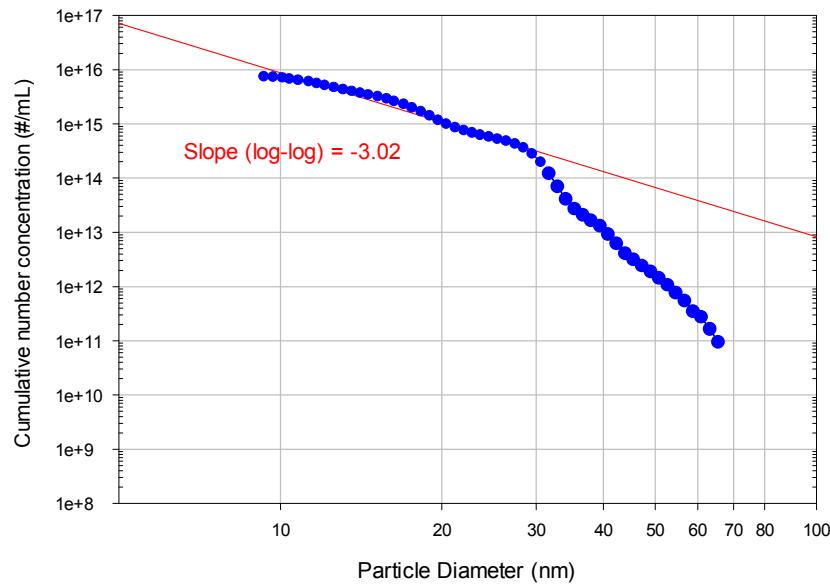
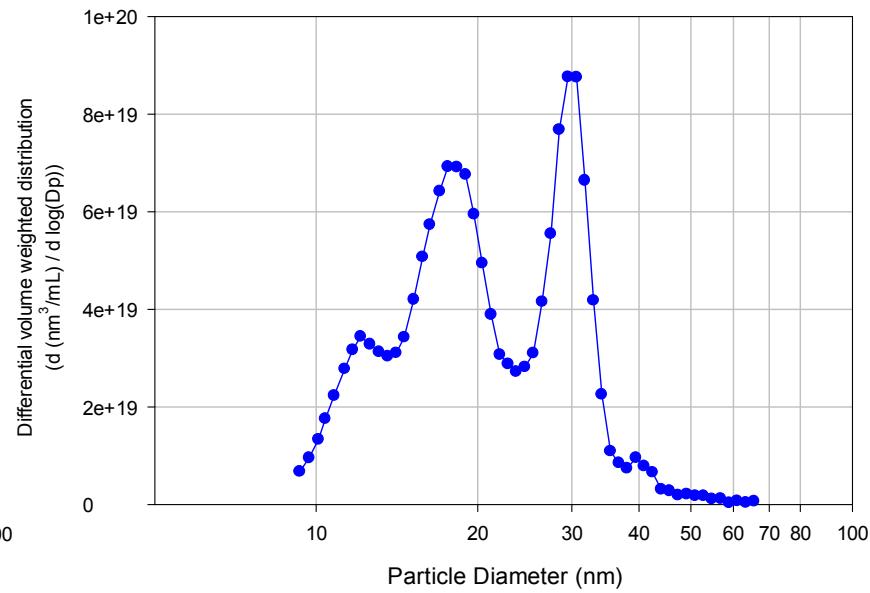
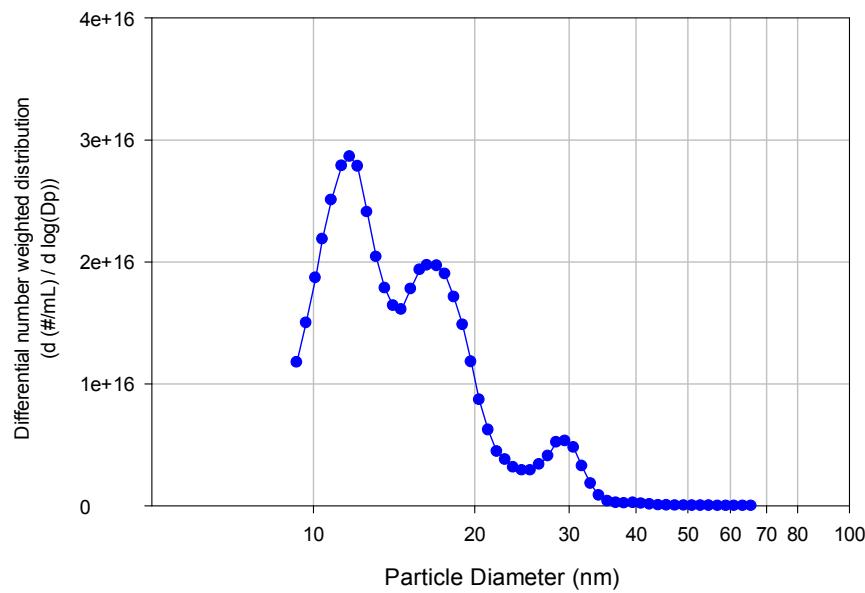


Poly-dispersed PSL challenge solution



Polydispersed Silica Challenge Solution

12, 18 and 28 nm colloidal silica



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Available materials and sizes

- Polystyrene latex (NIST traceable)
 - 20, 30, 40, 50, 60, 70, 80, 100 nm
 - 20 to 125 poly
 - 20 to 300 poly
- Colloidal silica
 - 10, 20, 30, 50, 70, 100 nm
 - 10/20/30 nm poly-silica
- Custom materials and sizes less than 500nm as requested



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Summary

- Technology is now available to measure the complete particle size distribution (size and concentration) of nanoparticles in liquids.
- The preparation of consistent PSL standards, with significant shelf life, has lead to the development of a more accurate calibration for optical particle counters.
- The ability to accurately measure the particle size distribution of colloidal suspensions allows for preparation of non-PSL concentration standards for particles smaller than 100 nm.
- A unique polydispersed PSL standard, comprising a mixture of 16 different PSL sizes (a PSL “cocktail”), offers new opportunities for the characterization of sub-100 nm pore size filters.



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