Nanoparticle Characterization Introduction: DLS, Diffraction, and NTA

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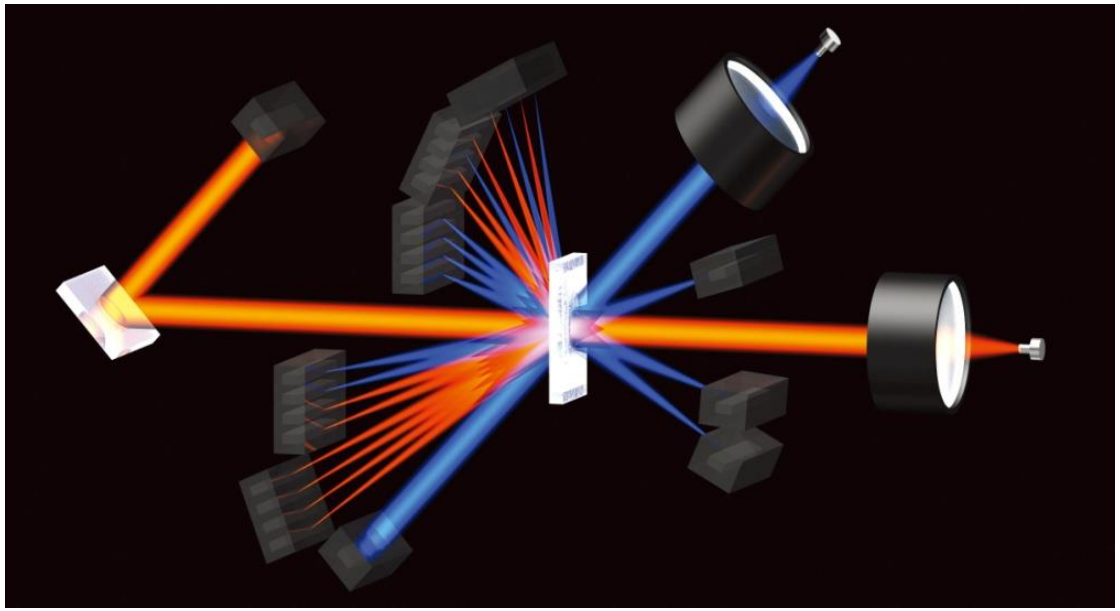
Outline

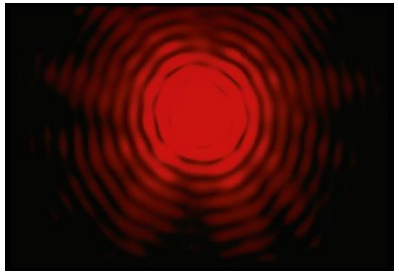
Overview of Techniques

- **Laser Diffraction**
- **Dynamic Light Scattering**
- **Nanoparticle Tracking Analysis and Multiple Lasers**

Laser Diffraction

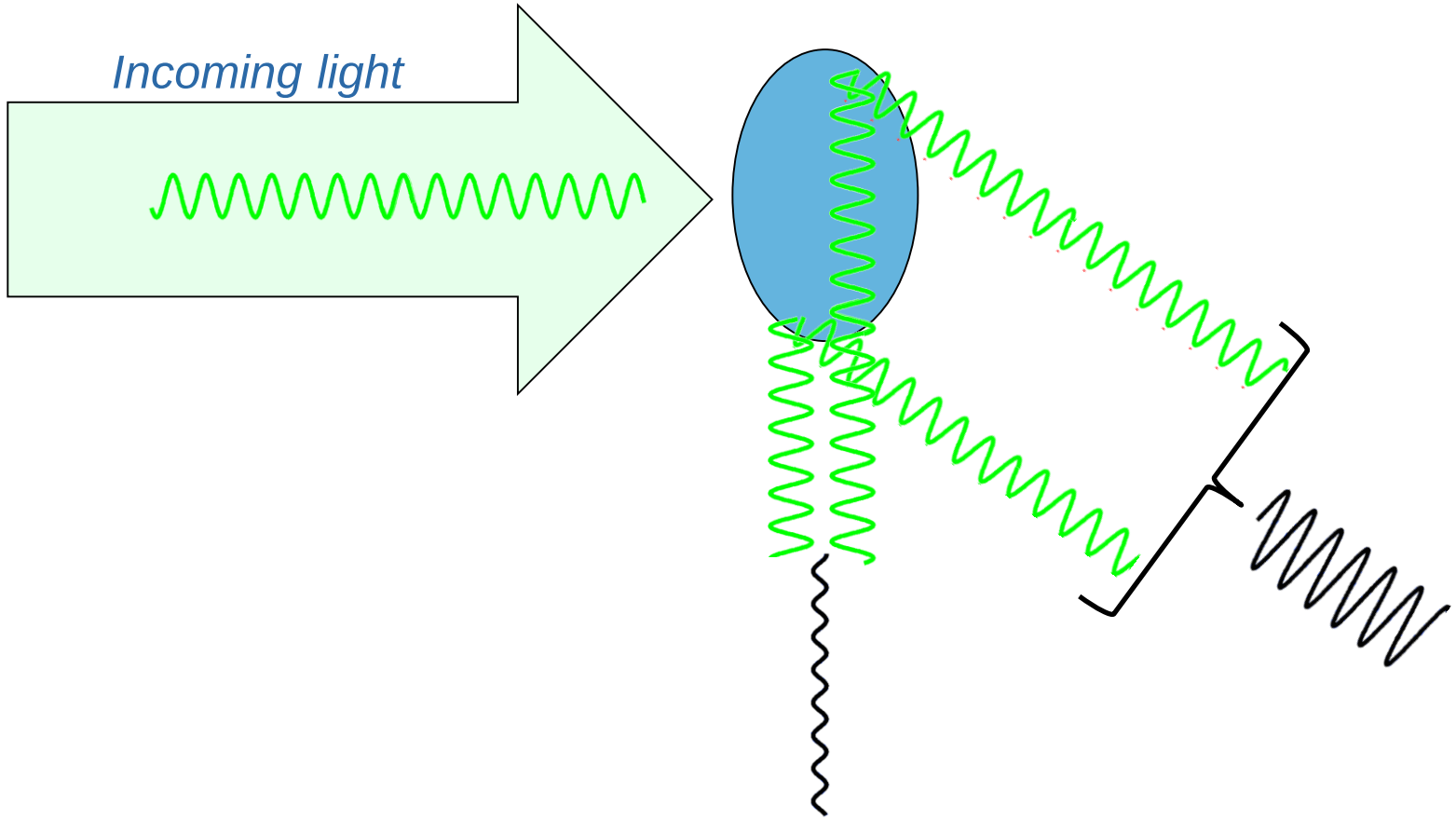
- Converts scattered light to particle size distribution
- Quick, repeatable
- Powders, suspensions
- Most common technique





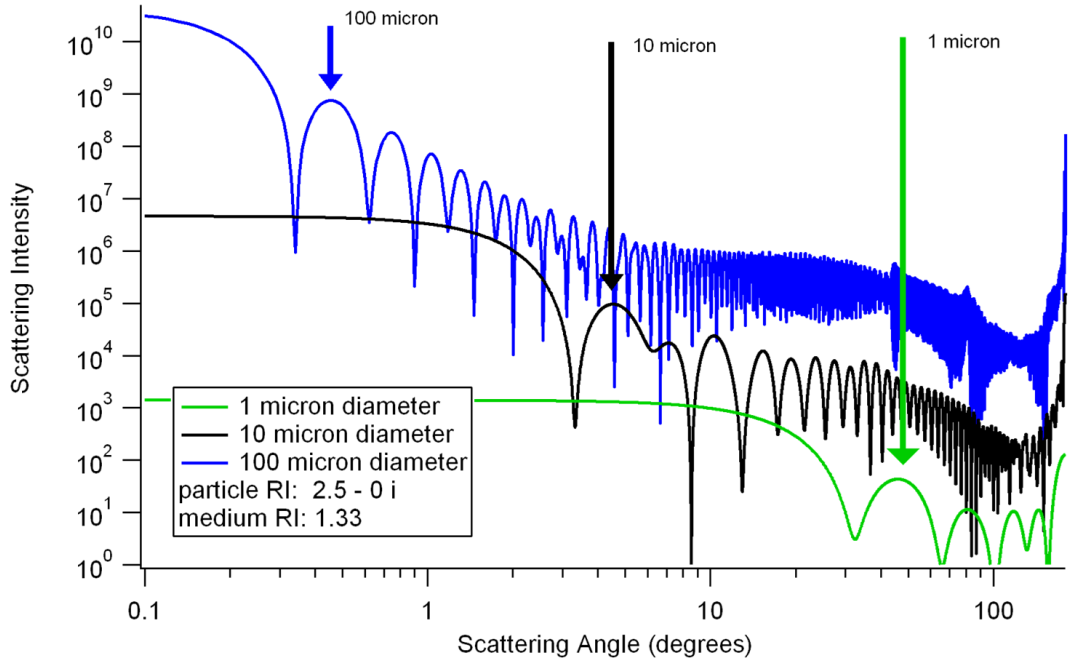
Path Length Difference

Diffraction effects arise due to scattering from various points in the particle (and, in the large particle limit only the edges)



Interpreting Scattering Data

- Scattering data typically cannot be inverted to find particle shape.
- We use optical models to interpret data and understand our experiments.
- Modern systems use particle refractive index in a 3-D calculation (Mie Theory) that includes behavior of light inside of particles.



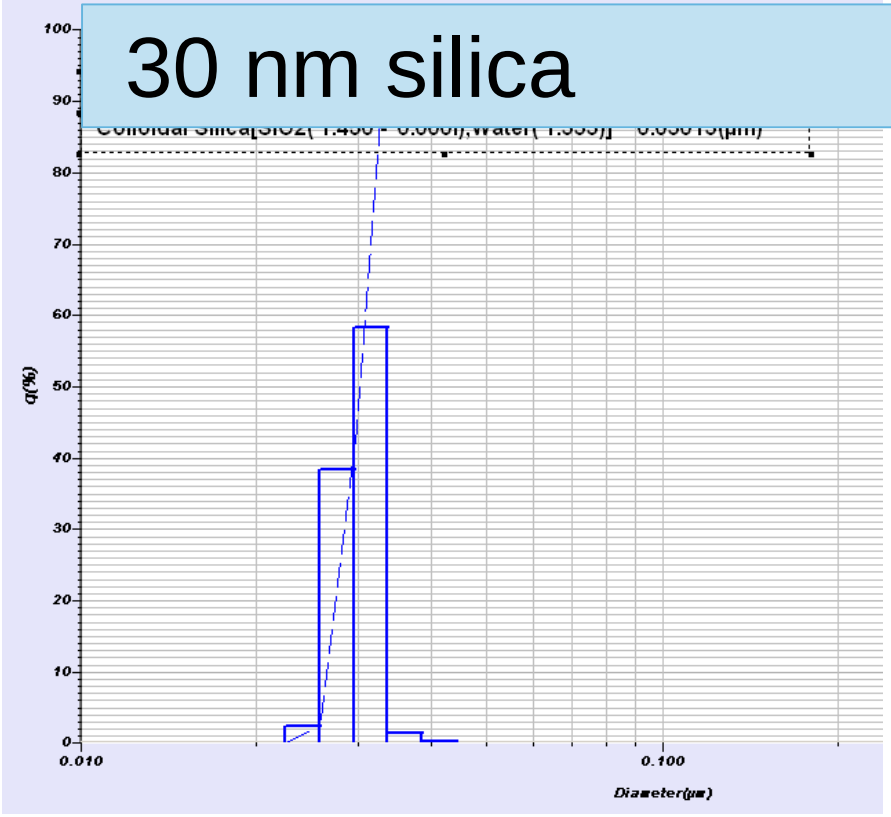
Fine particles: silica and latex

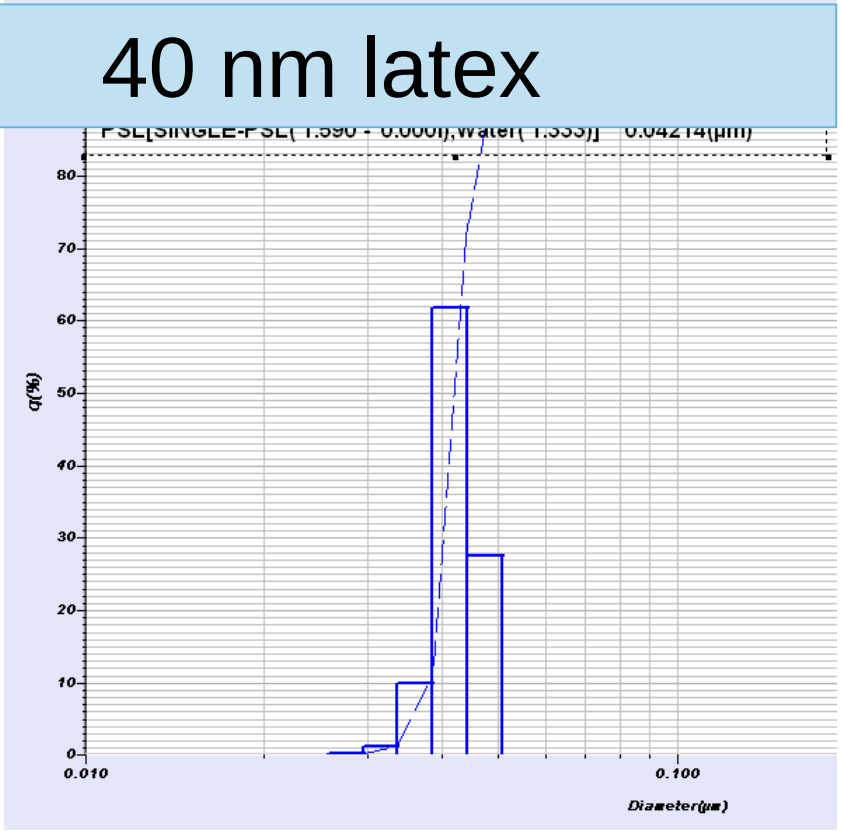
S.P.Area	:	2.0183E+6(cm²/cm³)
Mean Size	:	0.02990(µm)
Variance	:	5.0313E-6(µm²)
Median Size	:	0.03013(µm)
Mode Size	:	0.0302(µm)
Skewness	:	-0.2901

S.P.Area	:	1.4253E+6(cm²/cm³)
Mean Size	:	0.04241(µm)
Variance	:	1.2759E-5(µm²)
Median Size	:	0.04214(µm)
Mode Size	:	0.0422(µm)
Skewness	:	-0.1514

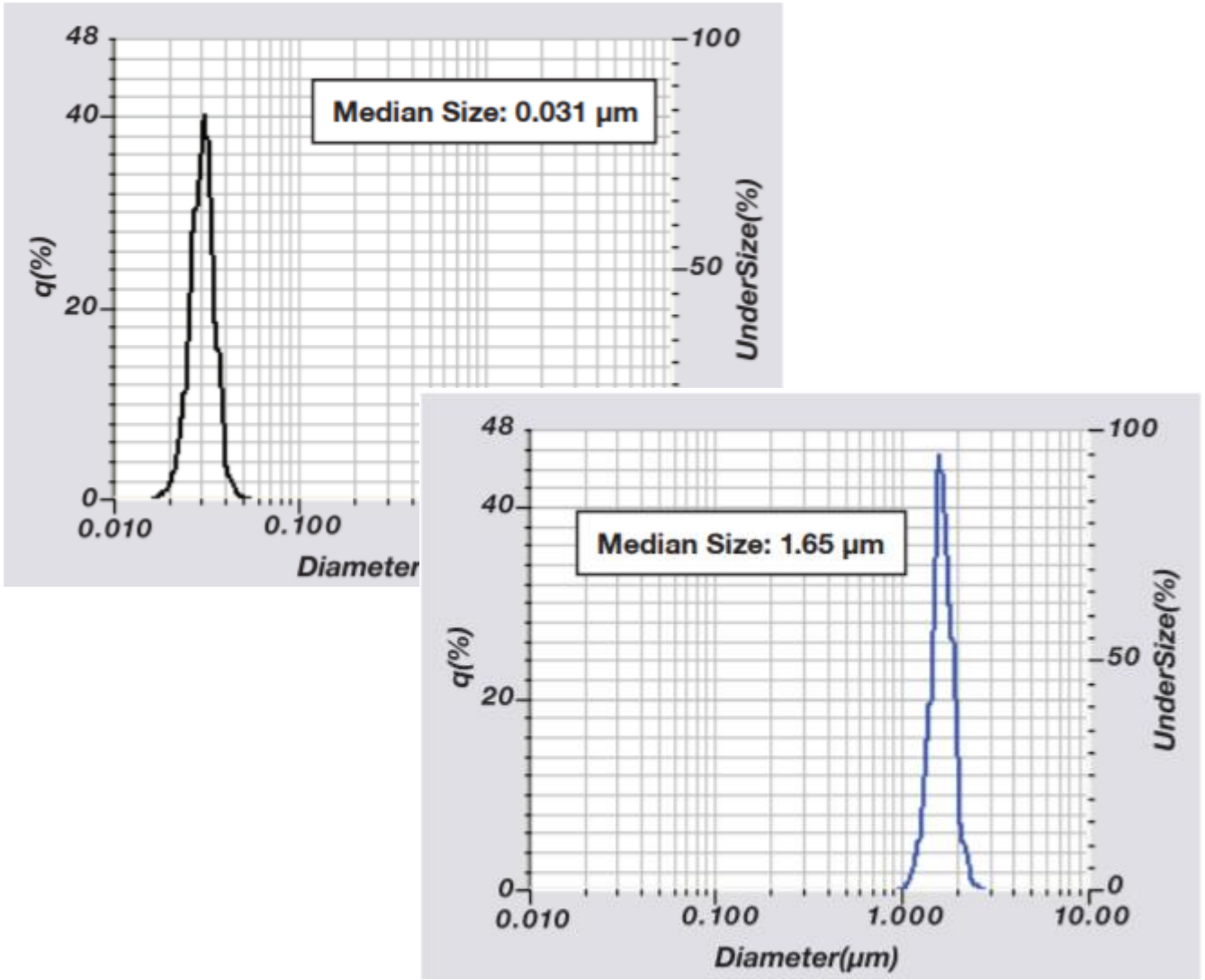
30 nm silica

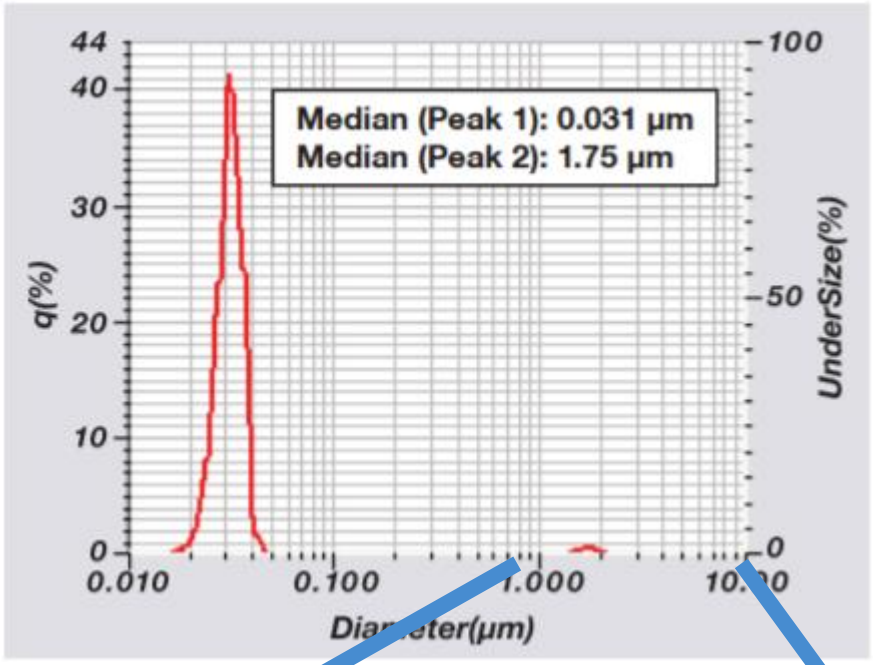
40 nm latex

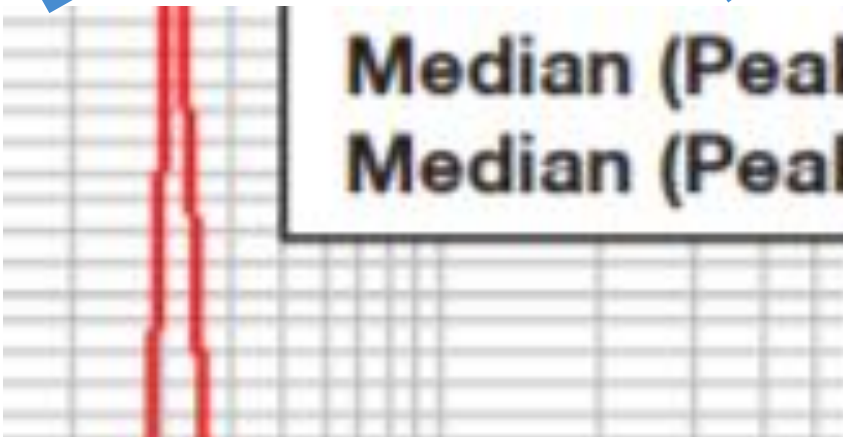




Finding large particle impurities







Instrument to instrument variation

4 instruments
(real sample)

	Dmean	D5	D10	D50	D90	D95
Average (nm)	155	112	119	152	193	208
Std. Dev. (nm)	0.8	0.8	0.7	1.0	1.1	0.7
CV (%)	0.5	0.7	0.6	0.6	0.6	0.3

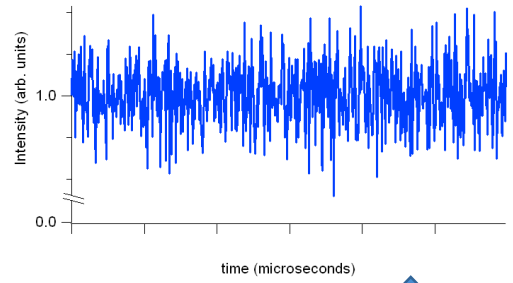
Figure 8: Instrument to instrument variation across four LA-950 systems for Formulation 1.

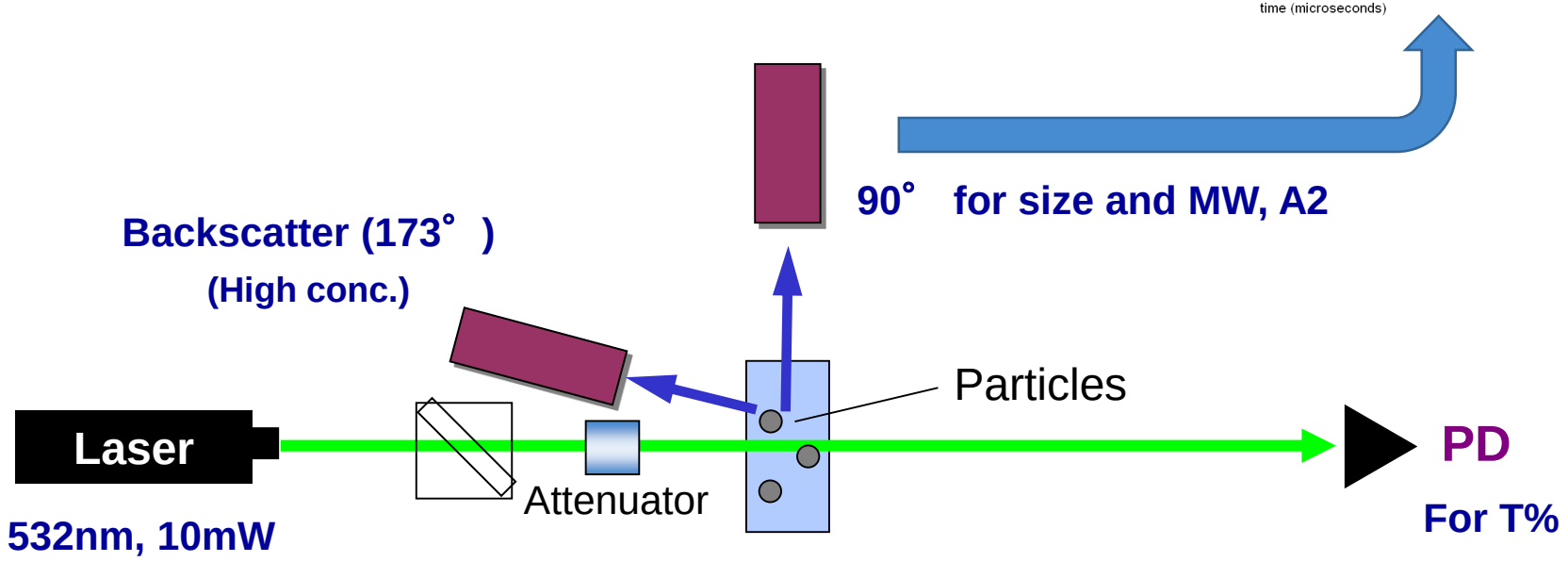
	Dmean	D5	D10	D50	D90	D95
Average (nm)	193	136	147	187	247	264
Std. Dev (nm)	1.5	0.5	0.4	0.6	0.4	1.1
CV (%)	0.8	0.4	0.3	0.3	0.2	0.4

Figure 9: Instrument to instrument variation across four LA-950 systems for Formulation 2.

DLS (Dynamic Light Scattering)

Use scattering as a function of time to determine size and size distribution

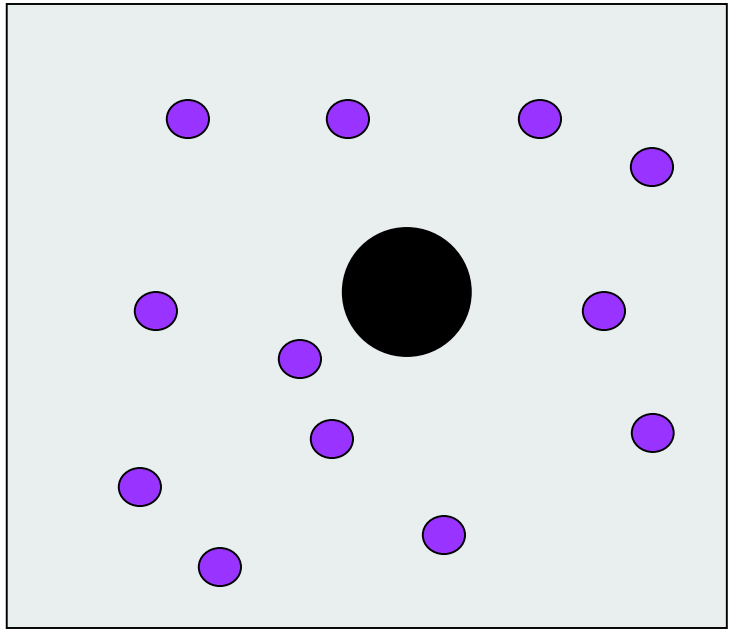




Particles moving due to Brownian motion

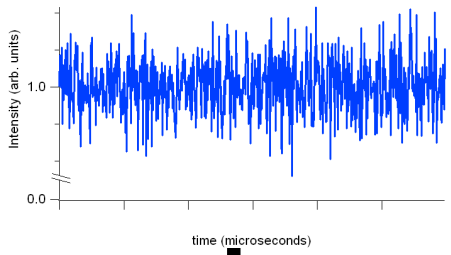
Brownian Motion

Particles in suspension undergo
Brownian motion (random thermal motion).



- Brownian Motion
  - Random
  - Related to Size
  - Related to viscosity
  - Related to temperature

Hydrodynamic Diameter



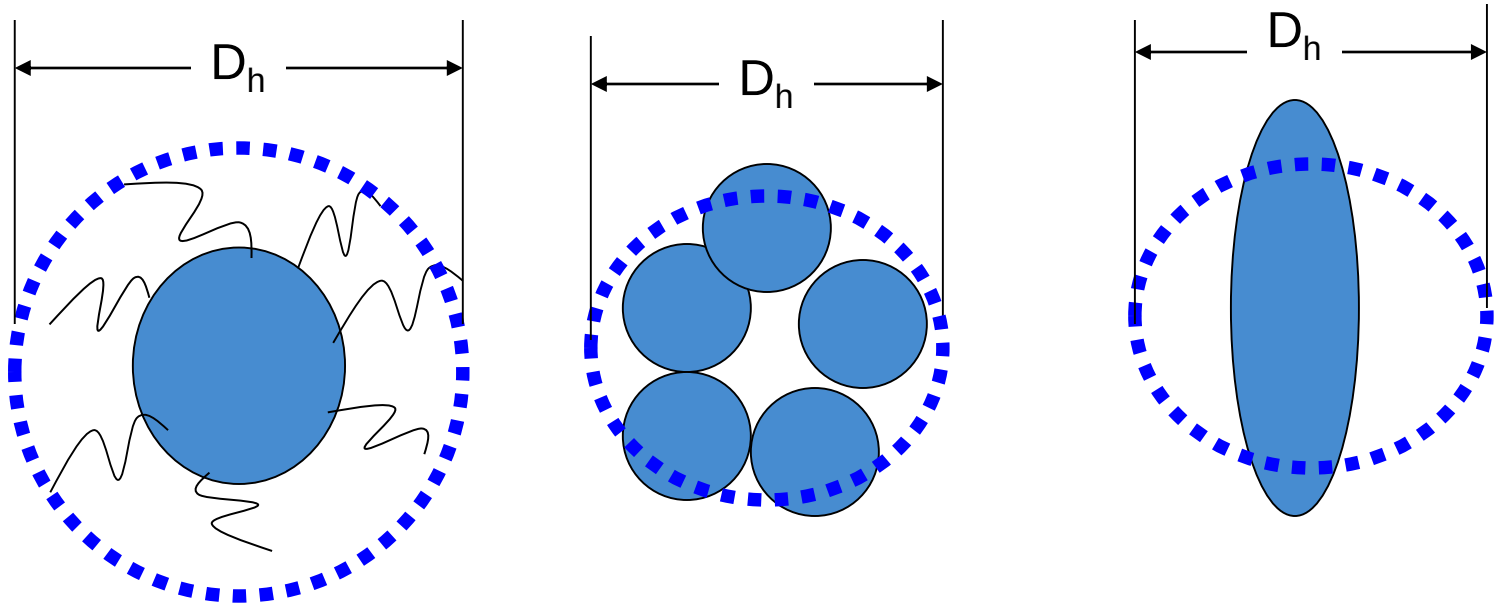
Then a miracle occurs …

$$D_h = \frac{k_B T}{3\pi\eta(T)D_m}$$

D_m diffusion coefficient
D_h hydrodynamic diameter
η viscosity
k_B Boltzman's constant

What is Hydrodynamic Size?

DLS gives the diameter of a sphere that moves (diffuses) the same way as your sample.



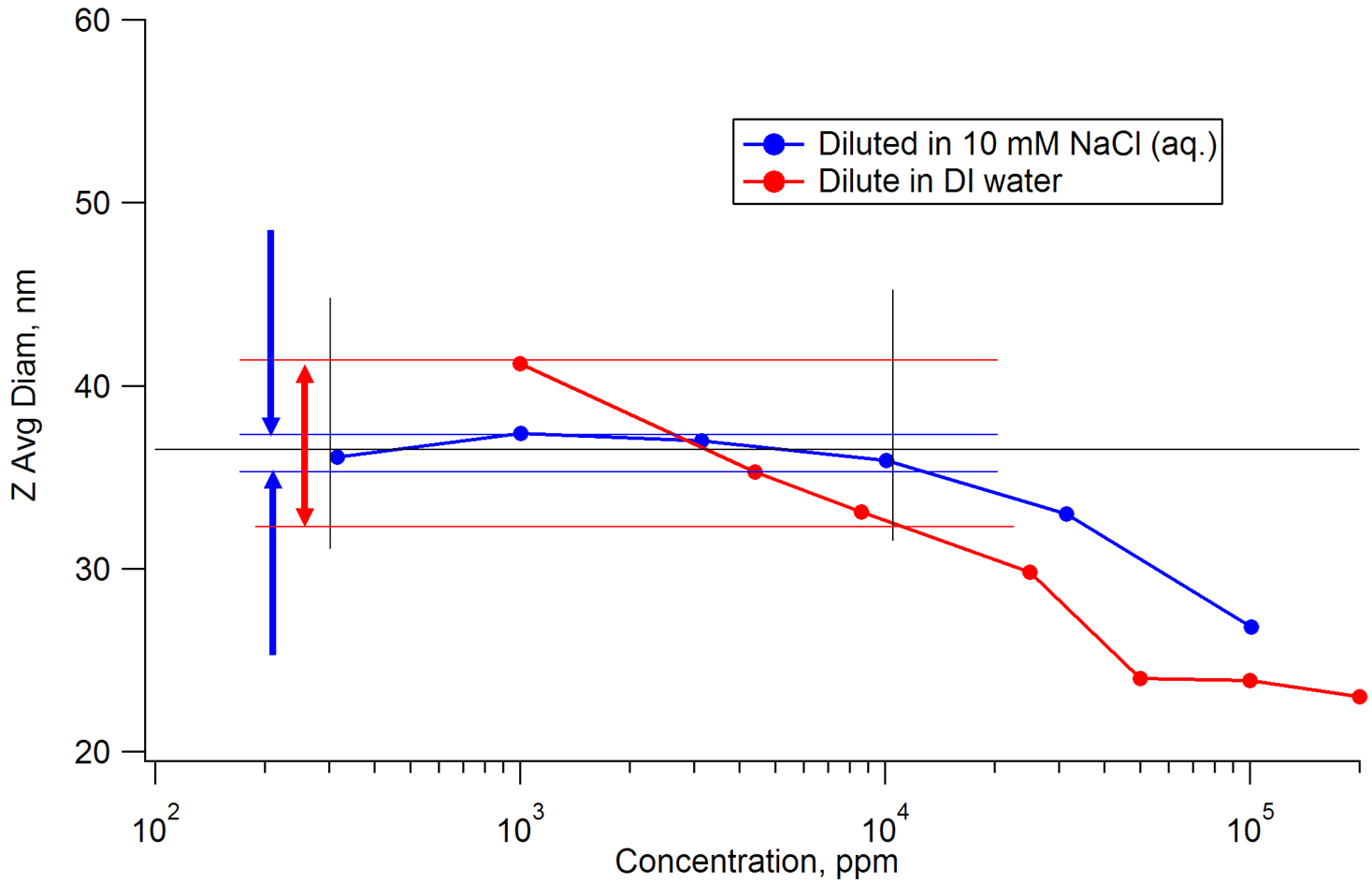
Lab to Lab comparison

Colloidal Silica

	Mean determined Z-average size (nm)	COV (%)
Dynamic Light Scattering with SZ-100, laboratory 1	34.4	0.7
Dynamic Light Scattering with SZ-100, laboratory 2	34.6	0.3

Effect of salt on measured size of silica

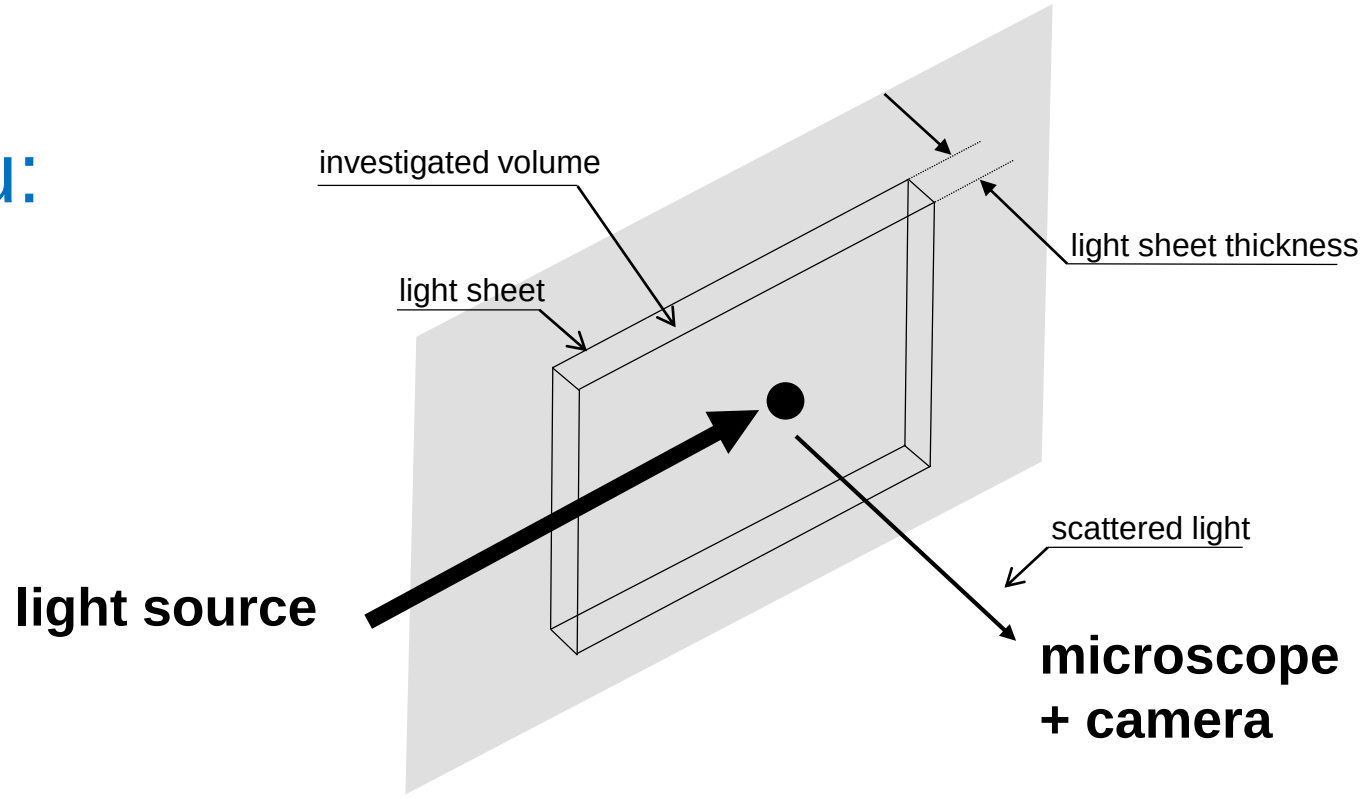
- Note that when we suppress effect of charges by adding salt, the effect of concentration is suppressed.
- Concentration effects are due to changes in particle motion, not just multiple scattering.



Nanoparticle Tracking (NTA)

Particle by particle gives you:

- Detailed Distribution
- Particle Concentration

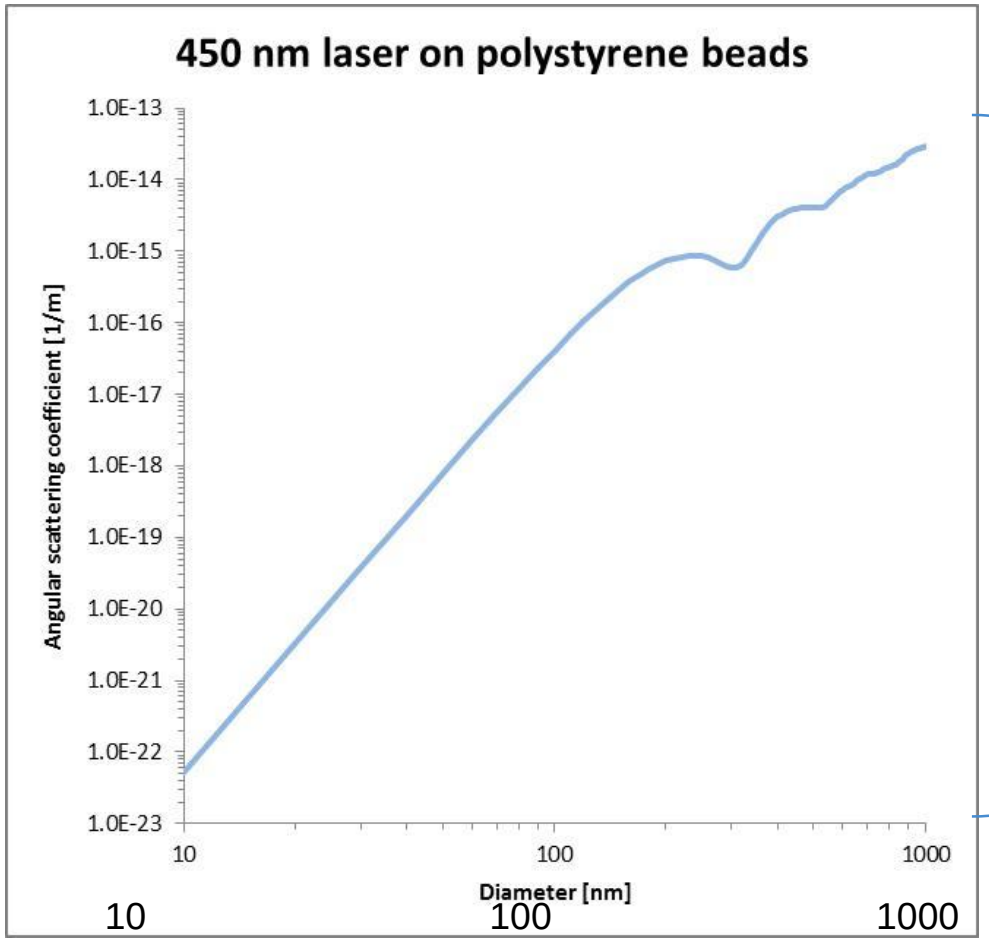


Problem: Intensity vs size

450 nm laser on polystyrene beads

Angular scattering coefficient [1/m]

1E-13



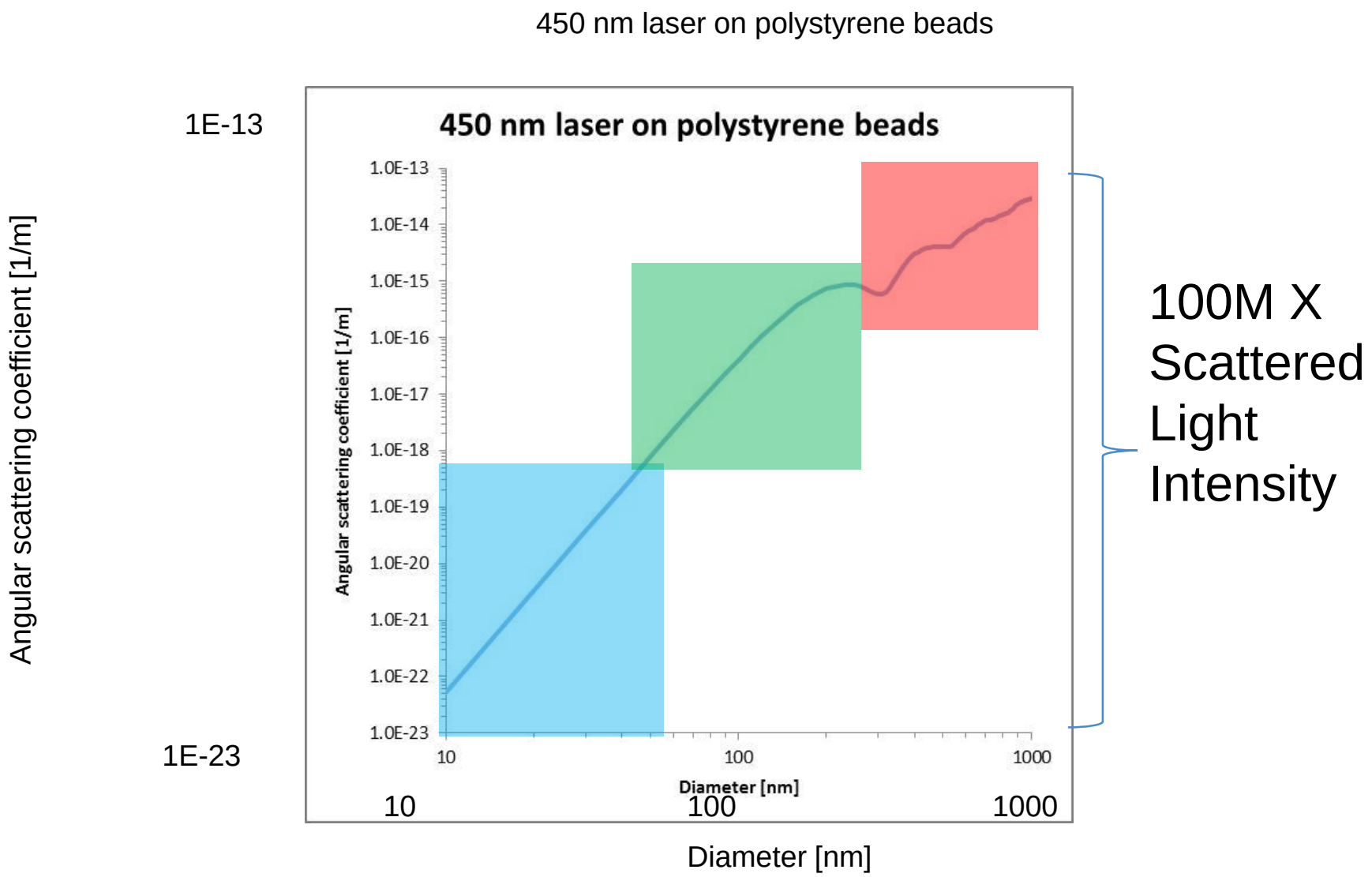
1E-23

10 100 1000

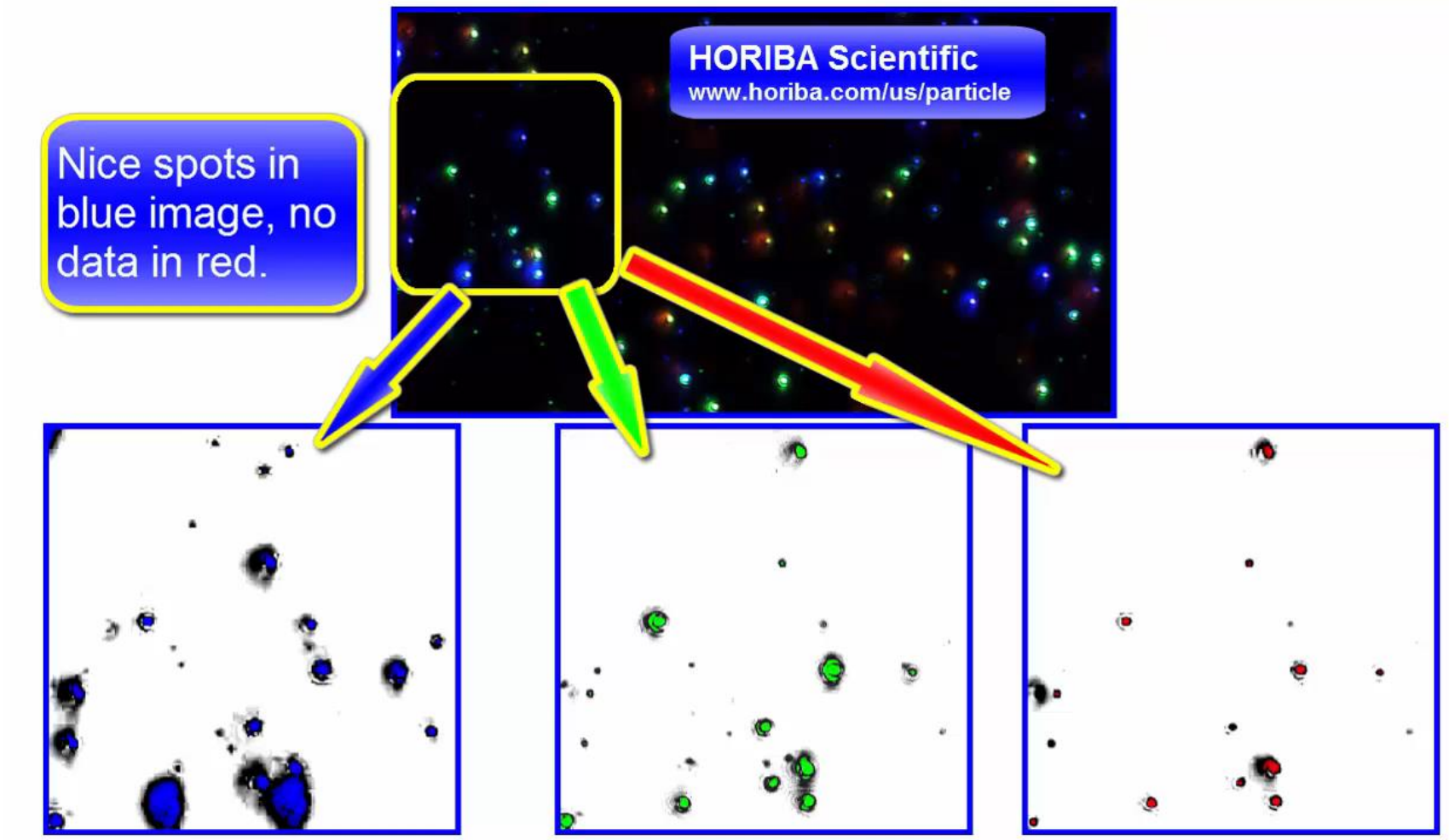
Diameter [nm]

100M X Scattered Light Intensity

Solution: Intensity vs size



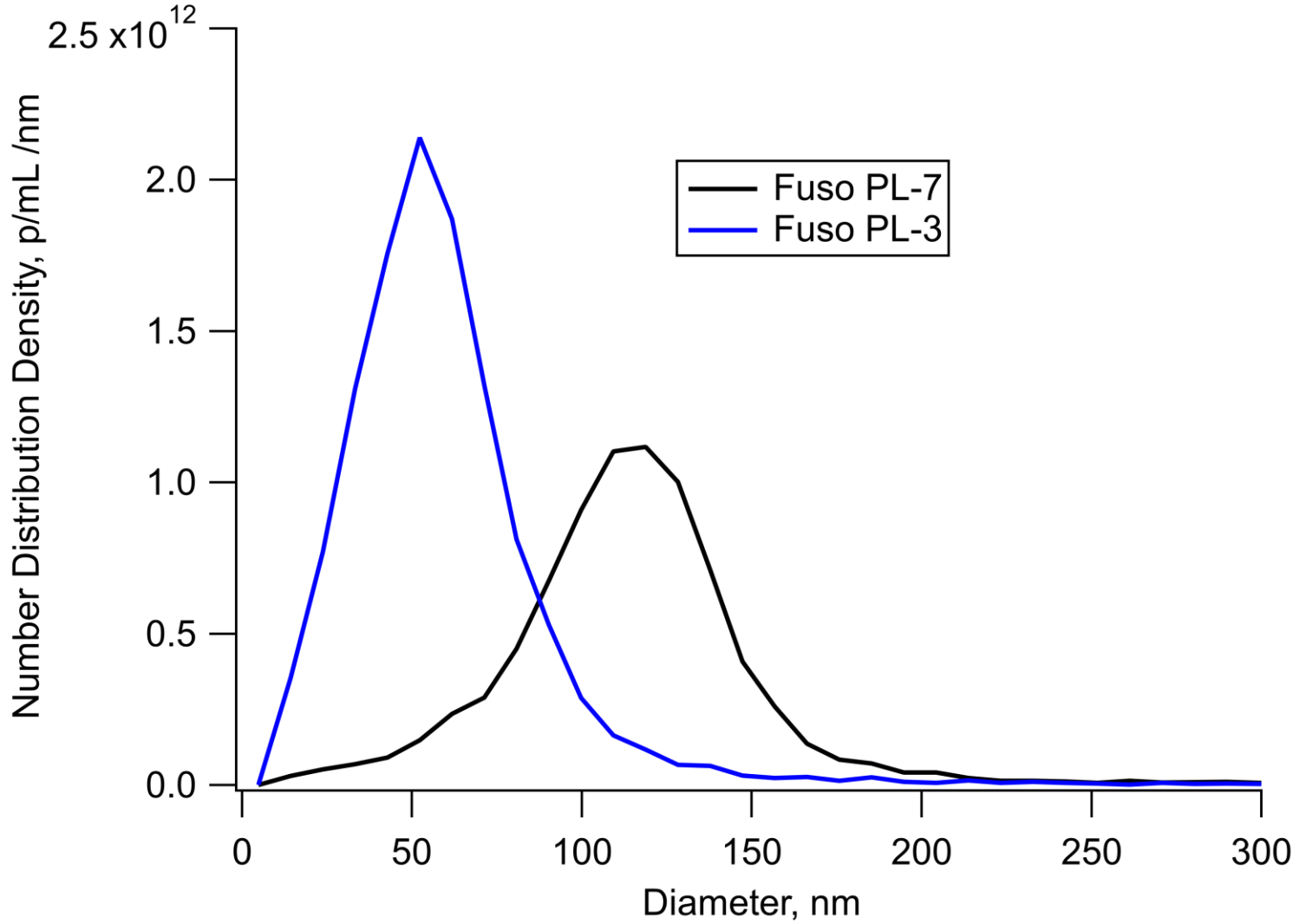
Why three colors?



Silica Particles by NTA

Number, not volume based distribution.

Particle concentration!



Key benefits of ViewSizer

- Individual particle method, not ensemble average
- Accurate PSD for polydisperse samples
- Concentration measured, not estimated
- Absolute method (no calibration needed)
- Particle visualization

Closing Comparison

Issue	Laser Diffraction	DLS	Multi-laser nanoparticle tracking
Large (>1 micron) particles in sample that need to be analyzed	++	--	-
Small quantity of sample	-	+	+
Smallest particles (<10~50 nm)	-	++	-
Speed	++	+	-
Nanoparticle Distribution	-	-	+
Analyze only tagged particles	--	--	+
Nanoparticle Concentration	--	--	+