

Particle Monitoring in Ultra Pure Water for Microelectronic Applications

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Agenda

- Motivation for Particle Monitoring
- Optical Particle Counting Fundamentals
- Instrument Specifications
- Counting Statistics
- Data Analysis and Interpretation



Particles: Integral part of ITRS Roadmap

Year of production	2005	2006	2007	2008	2009	2010	2011	2012	2013
DRAM half-pitch (nm)	80	70	65	57	50	45	40	36	32
Critical particle size (nm) [A]	40	35	33	29	25	22	20	18	16
Resistivity at 25°C(MΩ-cm)	18.2	18.2	18.2	18.2	18.2	18.2	18.2	18.2	18.2
Total oxidizable carbon (ppb) POE	<1	<1	<1	<1	<1	<1	<1	<1	<1
Bacteria (CFU/liter)	<1	<1	<1	<1	<1	<1	<1	<1	<1
Total silica (ppb) as SiO ₂	<0.5	<0.5	<0.5	<0.5	<0.5	<0.3	<0.3	<0.3	<0.3
Number of particles >0.05 μm (/ml) POE	<0.2	<1	<0.9	<0.8	<0.4	<0.3	<0.3	<0.3	<0.2
Dissolved oxygen (ppb) POE	<10	<10	<10	<10	<10	<10	<10	<10	<10
Dissolved nitrogen (ppm)	8-12	8-12	8-18	8-18	8-18	8-18	8-18	8-18	8-18
Critical metals (ppt, each)	<1	<1	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Other critical ions (ppt, each)	<50	<50	<50	<50	<50	<50	<50	<50	<50
Temperature stability (K)	±1	±1	±1	±1	±1	±1	±1	±1	±1
Temperature gradient in K/10 minutes POE for immersion lithography	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

Interim solutions are known

Manufacturable solutions are not known

Manufacturable solutions are known

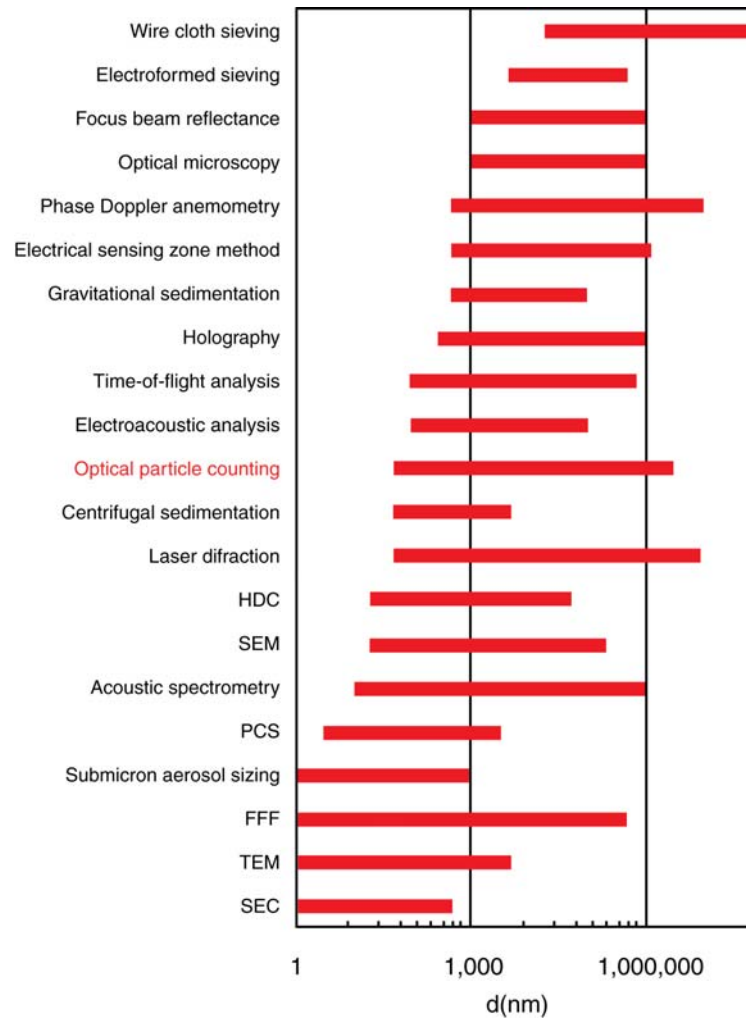


Ultra Pure Water

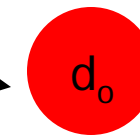
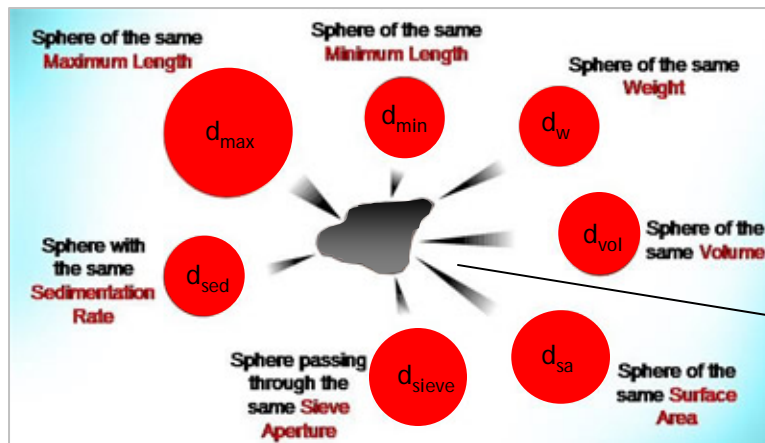
- Driving forces pushing the need for higher performance particle counters
 - 300 mm Fabs
 - Very few particles in newer UPW systems
 - <0.2 particles/ml (or 200/liter) larger than 0.05 microns
 - Particle failure of a UPW system has huge financial implications
 - Increased use
- Best monitoring technology for UPW is laser optical particle counting
 - Sensitivity
 - Reliability
 - Operator independent



Particle Sizing Methods



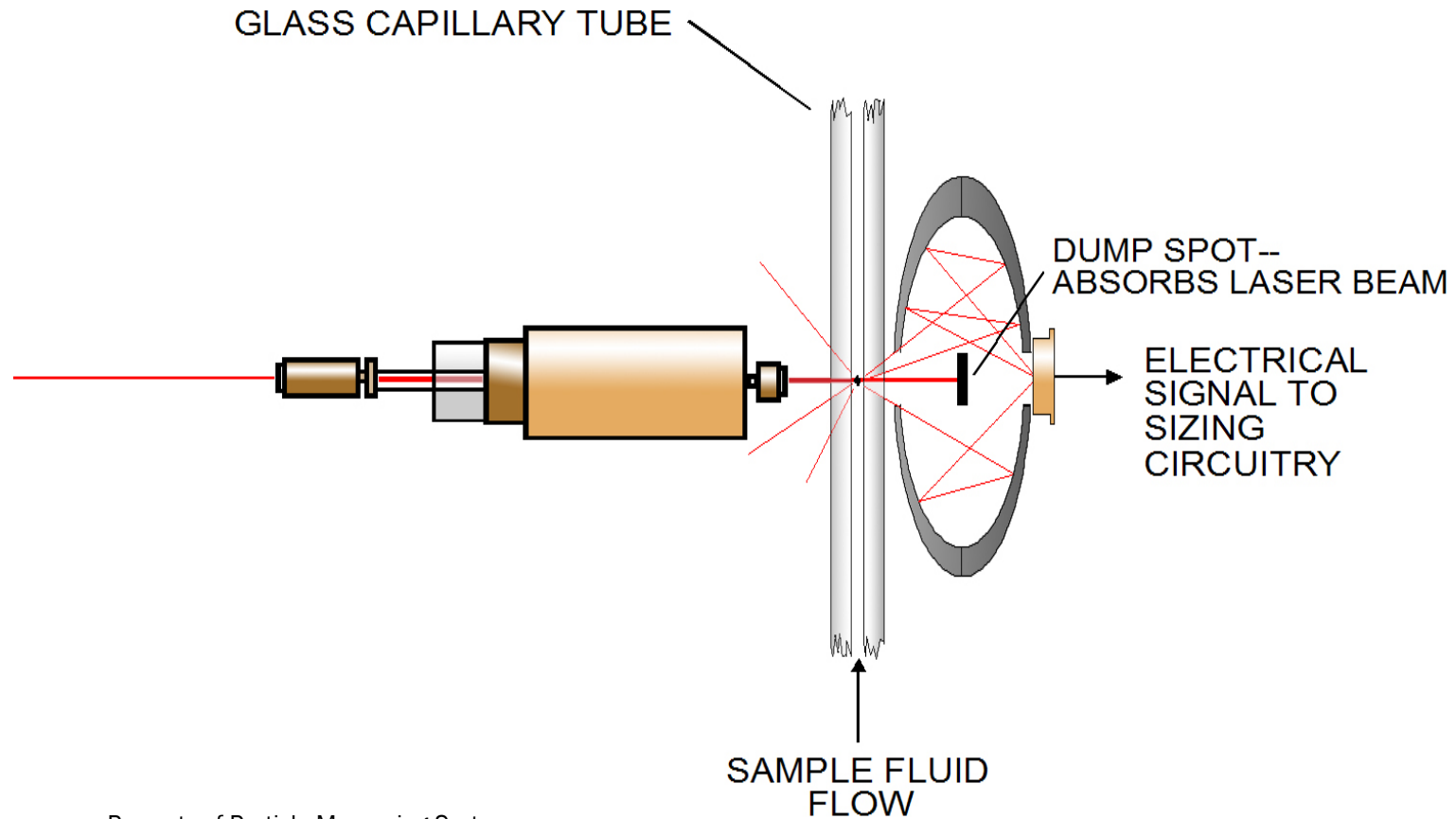
Spherical Equivalent Measurements



Sphere of the same
"optical equivalent"
(polystyrene latex)



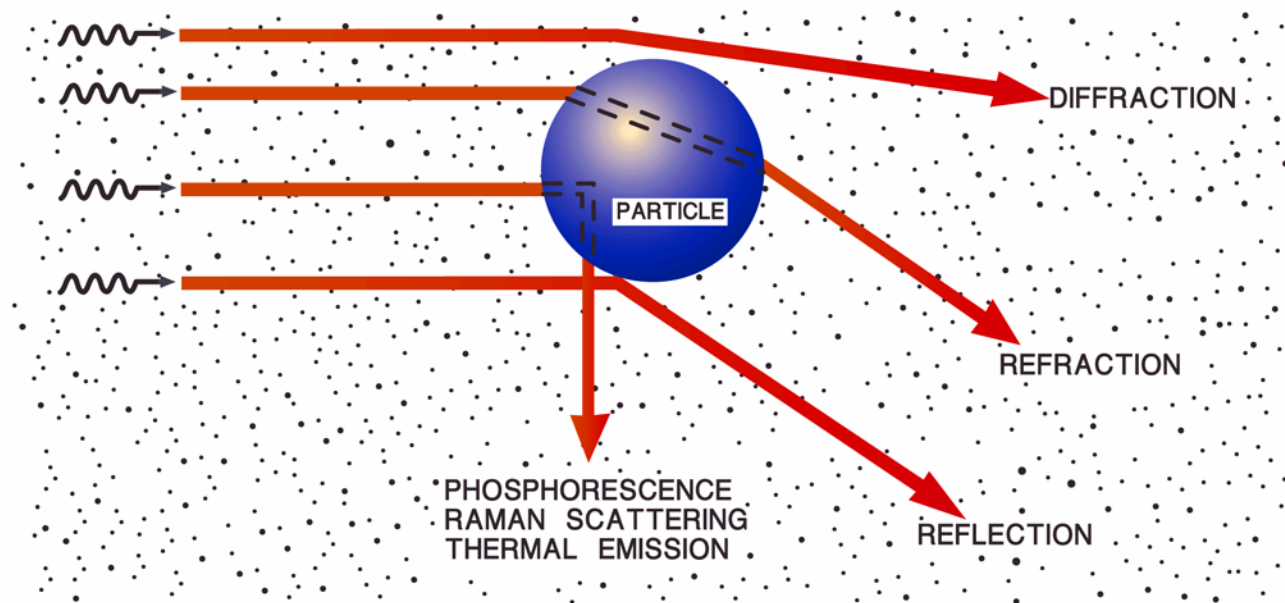
Liquid Particle Counter



Property of Particle Measuring Systems



Scattering Phenomenon

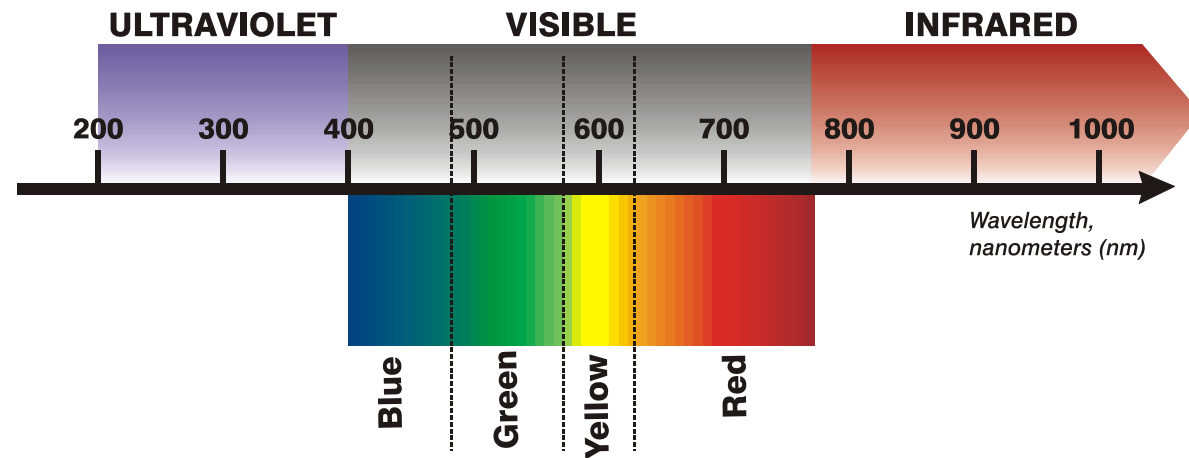


Property of Particle Measuring Systems



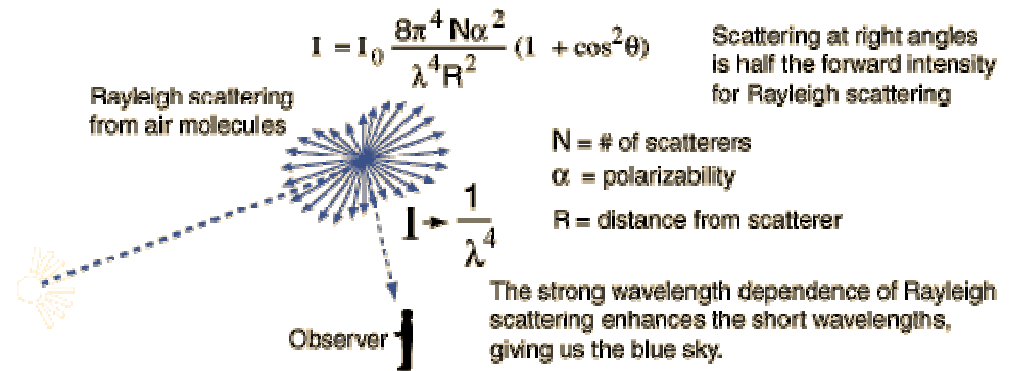
Scattering Domains

- Rayleigh Scattering
 - Particles much smaller than the light wavelength
- Lorenz-Mie Scattering
 - Particle sizes comparable to the light wavelength
- Geometric Scattering
 - Particles much larger than the light wavelength



Rayleigh Scattering

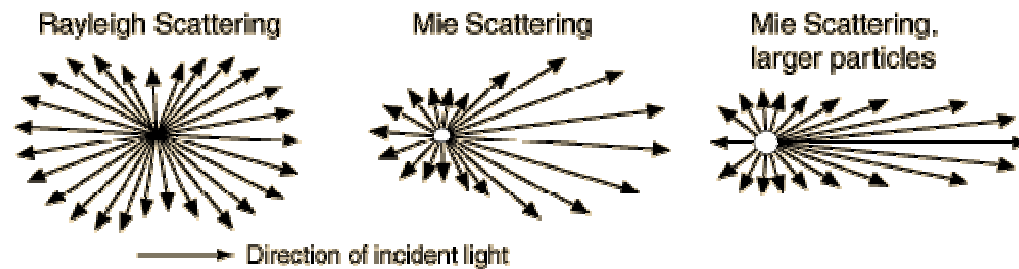
$$I_s \sim \lambda^2 f(\alpha) = \lambda^2 \alpha^6 = \frac{d^6}{\lambda^4}$$



- Scattering intensity largely independent of particle shape
- Refraction dominates
- Symmetrical scattering (forward=backward)



Lorenz-Mie Scattering



$$I_s \sim \lambda^2 f(\alpha) = \lambda^2 \alpha^{(6 > n > 2)} = \frac{d^{x < 6}}{\lambda^{y < 4}} \text{ to } \frac{d^{x > 2}}{\lambda^{y > 0}}$$

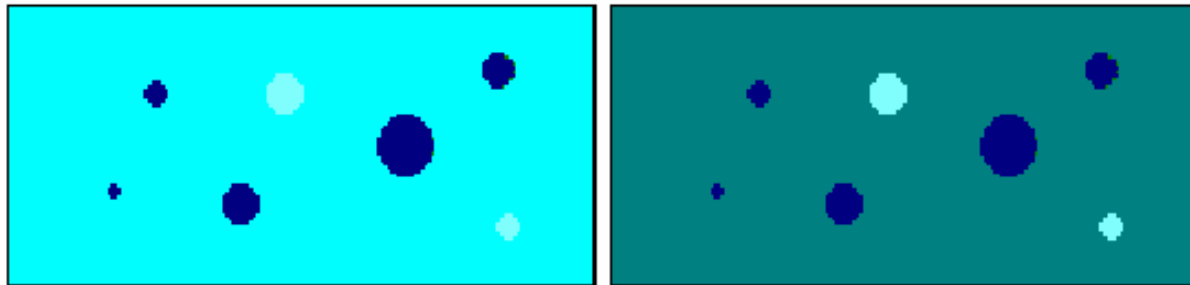
- Scattering intensity and angular distribution largely dependent of particle shape
- Intensity and polarization fluctuate as a function of scattering angle
- Asymmetrical scattering (forward \gg backward)
- Forward lobe mainly diffraction (becomes more concentrated)



Index of Refraction Component

- Scattering intensity is dependent on “IR Contrast”

$$IR_Contrast = \frac{Particulate_IR}{Media_IR}$$



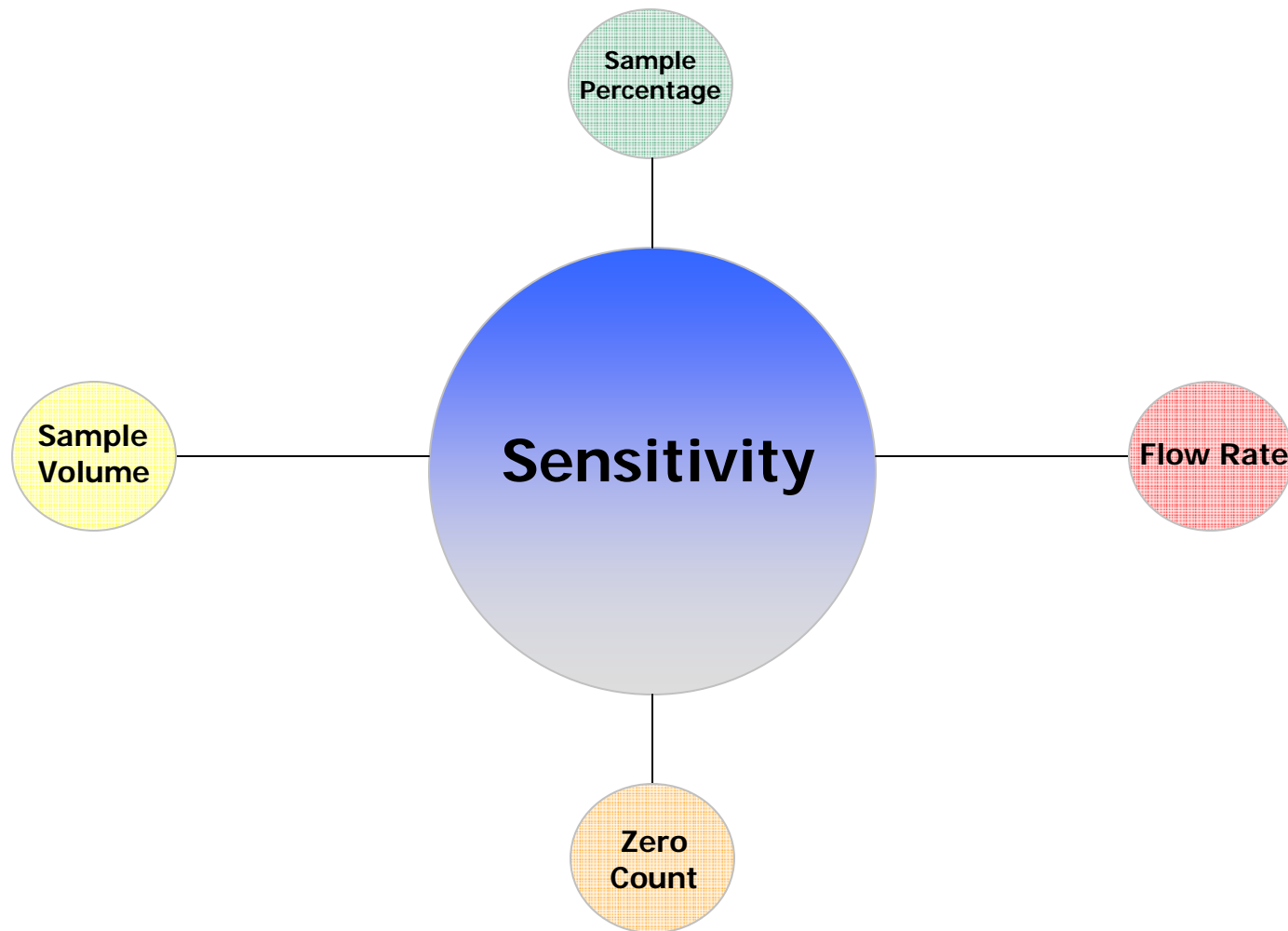
Index Of Refraction

- Refractive Indices of typical particles and various sample media ($\lambda = 633 \text{ nm}$)

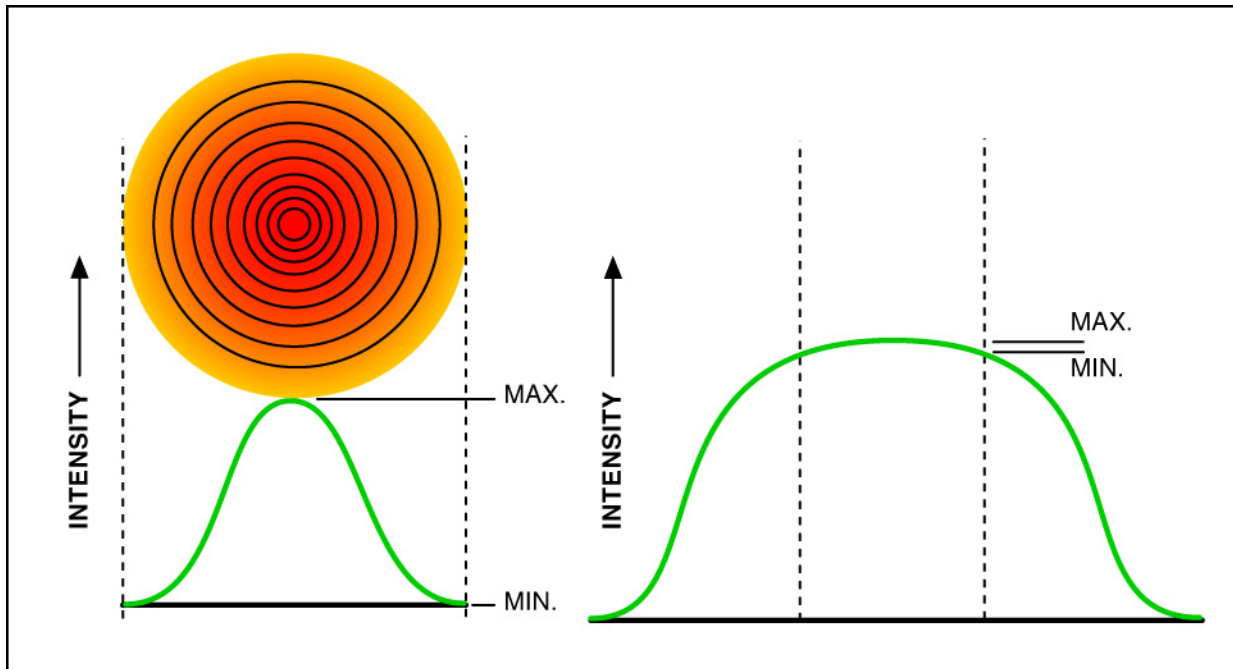
Particulate Material	Refractive Index
<i>Silicon Dioxide (Silica)</i>	1.45
<i>Silicon</i>	3.9
<i>Polystyrene Latex (PSL)</i>	1.59
<i>Steel</i>	2.5
<i>Copper</i>	0.45
<i>Aluminum</i>	1.9
Sample Media	
<i>Water</i>	1.33
<i>Hydrofluoric Acid (50%)</i>	1.29
<i>Ammonium Hydroxide (29%)</i>	1.33
<i>Hydrochloric Acid (37%)</i>	1.41
<i>Sulfuric Acid (96%)</i>	1.46



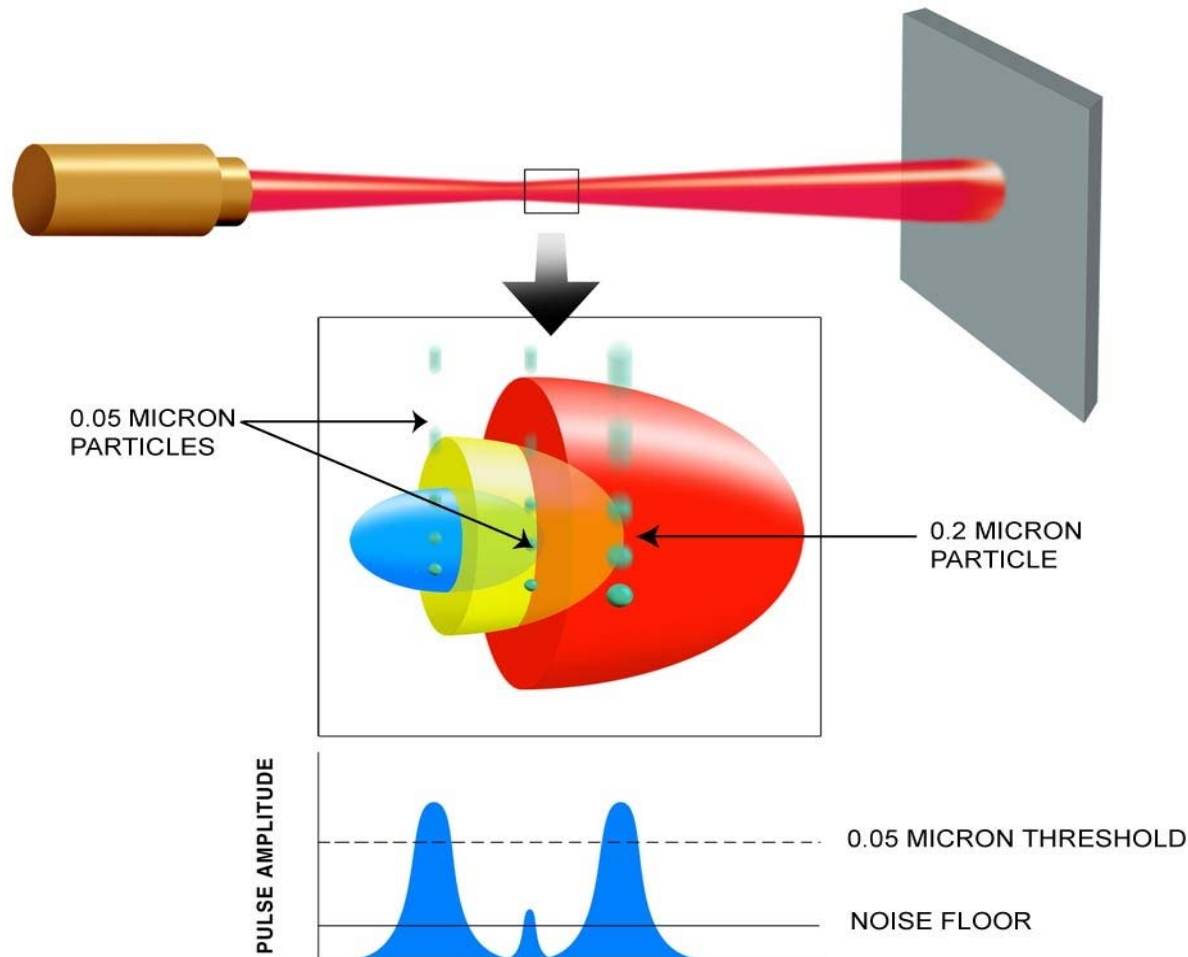
Instrument Considerations



Laser Intensity Profile



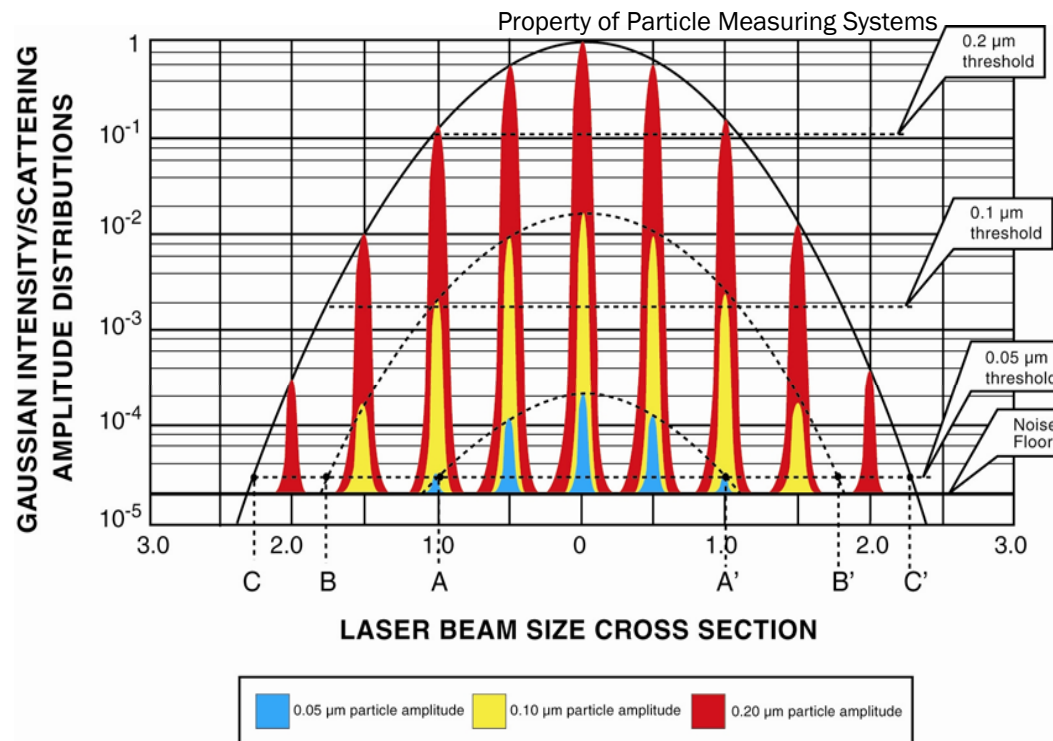
Sizing vs. Laser Beam Profile



Property of Particle Measuring Systems



Size Thresholds vs. Laser Beam Profile

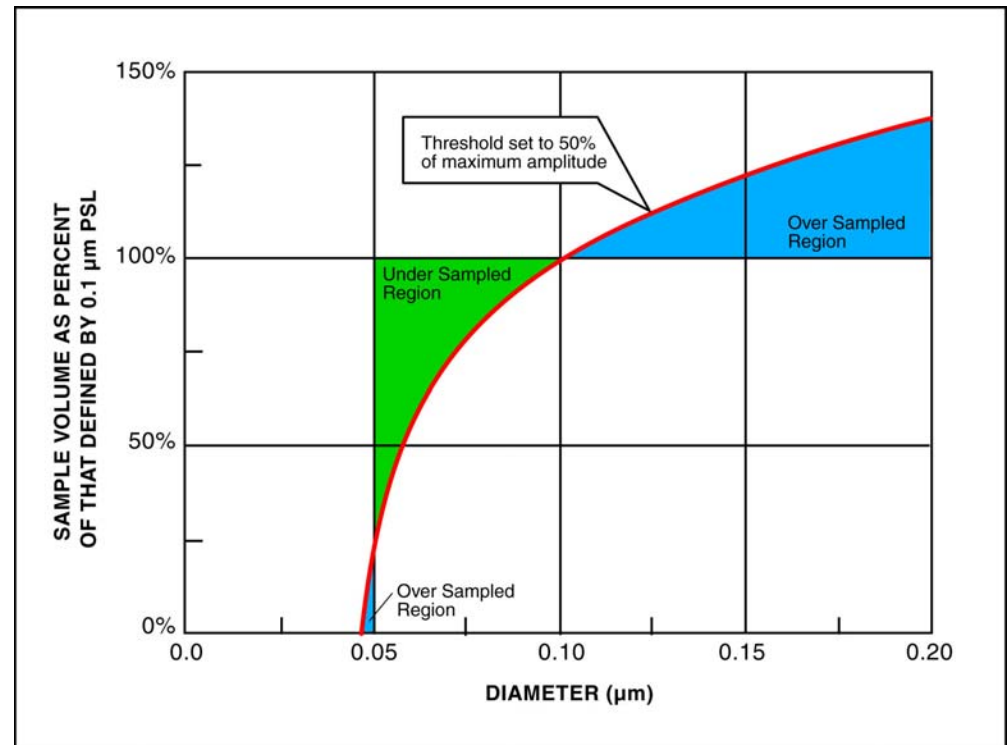


- Particles will only be sized most accurately – A to A'
- Large particles can be binned in the first channel
- 4x difference in particle size = 3,000X difference in signal



Sample Volume for Non-Volumetric Monitors

- Similar to volumetric, except a second over counting region exists
- Large particles are incorrectly sized in the first channel
- Over counting can be significant for poor OPC designs



Effect of Sample Volume

- Without measurement there is no control
 - Without data ...
 - Assume 100 particles/L > 0.05 μm

Particle Counter	Sample Volume	Time to measure 1 Liter fluid
Ultra DI® 50	3.75 mL/min	4.4 hrs
HSLIS M50e	0.25 mL/min	2.8 days
Competitor	0.1 mL/min	6.9 days

Particle Counter	Sample Volume	Time to measure 1 particle (min)	Time to measure 20 particles (hr)
Ultra DI® 50	3.75 mL/min	2.7	.9
HSLIS M50e	0.25 mL/min	40	13.3
Competitor	0.1 mL/min	100	33.3



Counting Statistics

- Poisson statistics are used when analyzing discrete events that are considered to be randomly distributed
- Particles are discrete events randomly distributed in time and space, so “particle counting” is described by a Poisson distribution
- Poisson statistics
 - Mean = λ
 - Variance = λ
 - Standard Deviation = $\sqrt{\lambda}$

Implications: Variability in particle count measurements are largely dependent on the number of particles counted per unit time



Example

- Assume you have 100 particles per liter at $> 0.05 \mu\text{m}$
- Measured with Ultra DI[®] 50
- Control limit at 3 S.D. above average

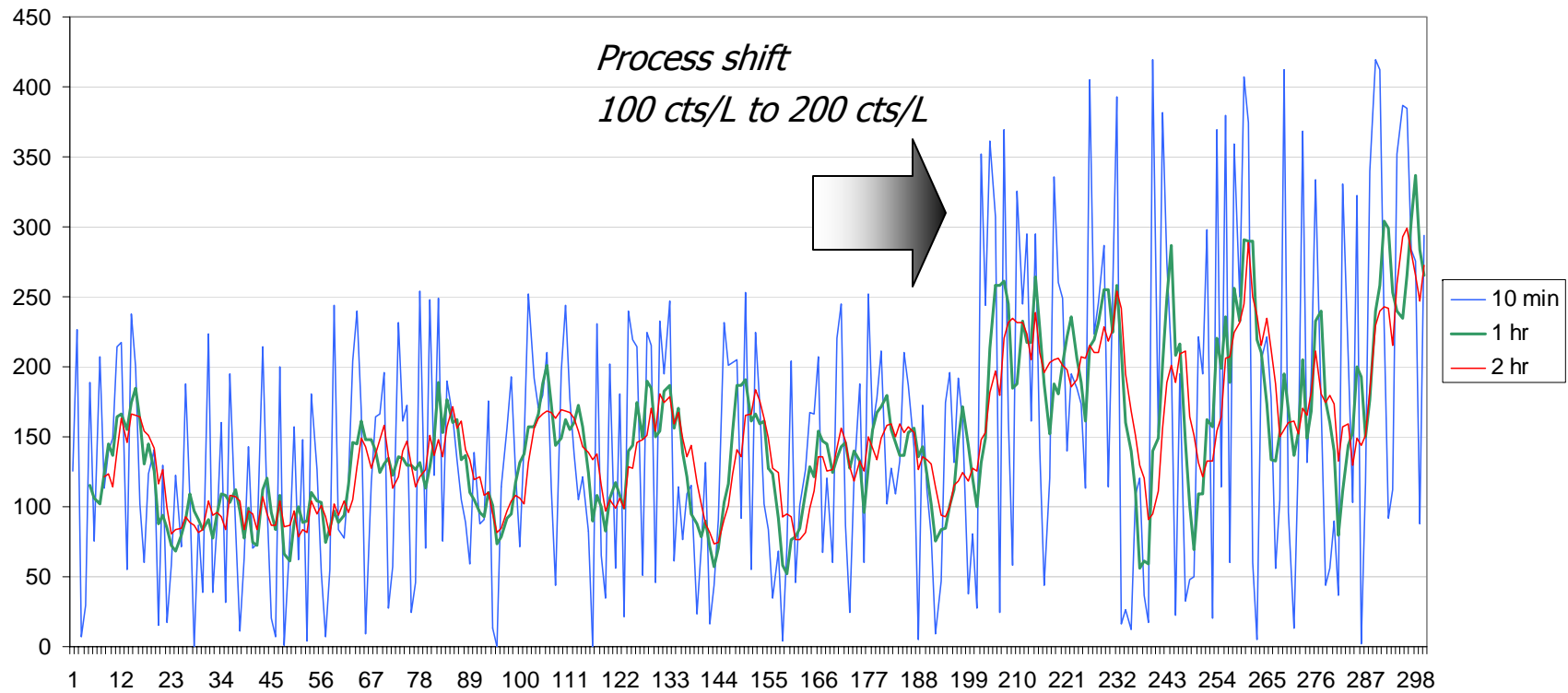


Sample Interval	10 min	20 min	1 hr	2 hr	4 hr
Control Limit	250	210	160	140	130



Graphical Representation

Effect of Sample Interval on Counting (cts/L vs time)



Statistics Summary

- Total number of particles measured can be very low
 - High variability in results occurs when only a small volume is measured and few particles counted
 - Resulting low quality data provides a poor measure of system performance
- Counting statistics often is the performance limiter
- The more particles counted per unit time:
 - More repeatable the measurement
 - Tighter control limits
 - Better understanding of process variations
 - Prevention of excursions



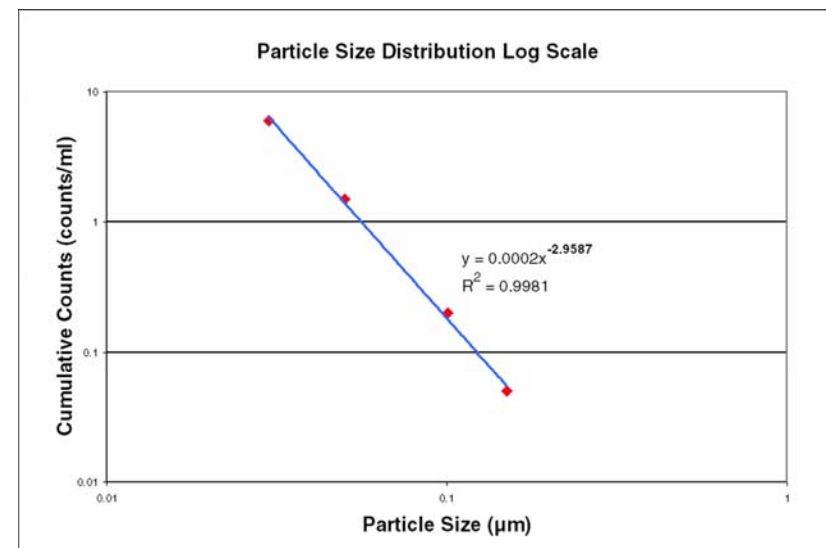
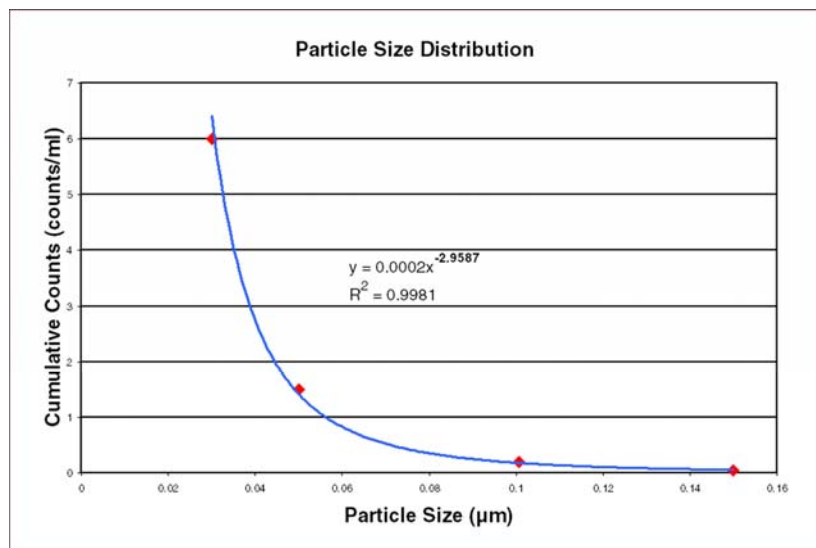
Zero Count Levels

- Counts in the absence of particles
- Ultra DI[®] 50 zero-count spec is < 50 cts/liter
- Assume zero particles
 - Zero count @ 50 per liter
 - Average 1 count every 5.33 min
 - 83 particles per liter 3 S.D. control limit with 120 min sample intervals
- Zero count affected by cosmic rays, electronic noise, etc.



PSD in Ultrapure Water

- Typical particle size distributions in UPW
 - $1/(\text{particle diameter})^3$ is typical for UPW
- More small particles than large particles



Impact of Size Sensitivity

- UPW system

- $1/(\text{diameter})^3$ distribution
- 1 particle/mL $>$ 0.05 μm

- How many particles $>$ 0.025 μm ?

- = (number of particles $>$ 0.05 μm) * $1/(\text{ratio of diameters})^3$
- $1 * 1/(.5)^3 = \underline{8 \text{ particles/mL} > 0.025 \mu\text{m}}$

- Rule of thumb

- If particle diameter decreases by 2x
- Number of particles/mL (rate) increases by 8x



Summary

- Particle monitoring is a key part of the ITRS roadmap
- Optical Particle Counters are based on “optical equivalent” measures of scattering to that of PSL’s
- Sensitivity is only one measure of instrument performance
- A critical metric of performance is total particles counted
- Larger sample volume provides more data and improved counting statistics
- Properly operating UPW systems follow a $1/\text{diameter}^3$ particle size distribution





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