



# Help! How Can I Trust My Particle Size Results?



**Ian Treviranus**

[ian.treviranus@horiba.com](mailto:ian.treviranus@horiba.com)

[www.horiba.com/particle](http://www.horiba.com/particle)

# What we'll talk about

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- **General concerns**
- Orthogonal techniques
- Confirming particles  $< 1$  micron
- Confirming particles  $> 1$  micron
- Q&A

# Starting point

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- Instrument is fully functional 
  - System verification webinar TR004
- Suitable sample for technology 
  - Counters are classic example
- Boot Camp webinars free on website 
  - Particle size basics, RI selection, sampling & dispersion advice, method development for maximum precision

# General concerns



- Is my distribution really this broad?
  - Light scattering broadens distributions
  - LS resolution < IA resolution
- How to handle multi-mode results?
  - Discuss how to test different peaks today
  - Use multi-modal report for better metrics
- Data matching
  - Different instruments or different techniques will provide different data



# Method validation

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- Have existing method, does it produce “good” data?
- Test robustness of method
  - Measurement length
  - ALWAYS! collect multiple samplings
    - Reproducibility > challenge than repeatability
    - Evaluate with COV of chosen metric(s)
- Check with orthogonal technique



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# Featured technologies



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## ■ **LA-950**

Laser Diffraction

## ■ **SZ-100**

Dynamic Light Scattering & Zeta Potential

## ■ **CAMSIZER & CAMSIZER XT**

Dynamic Image Analysis

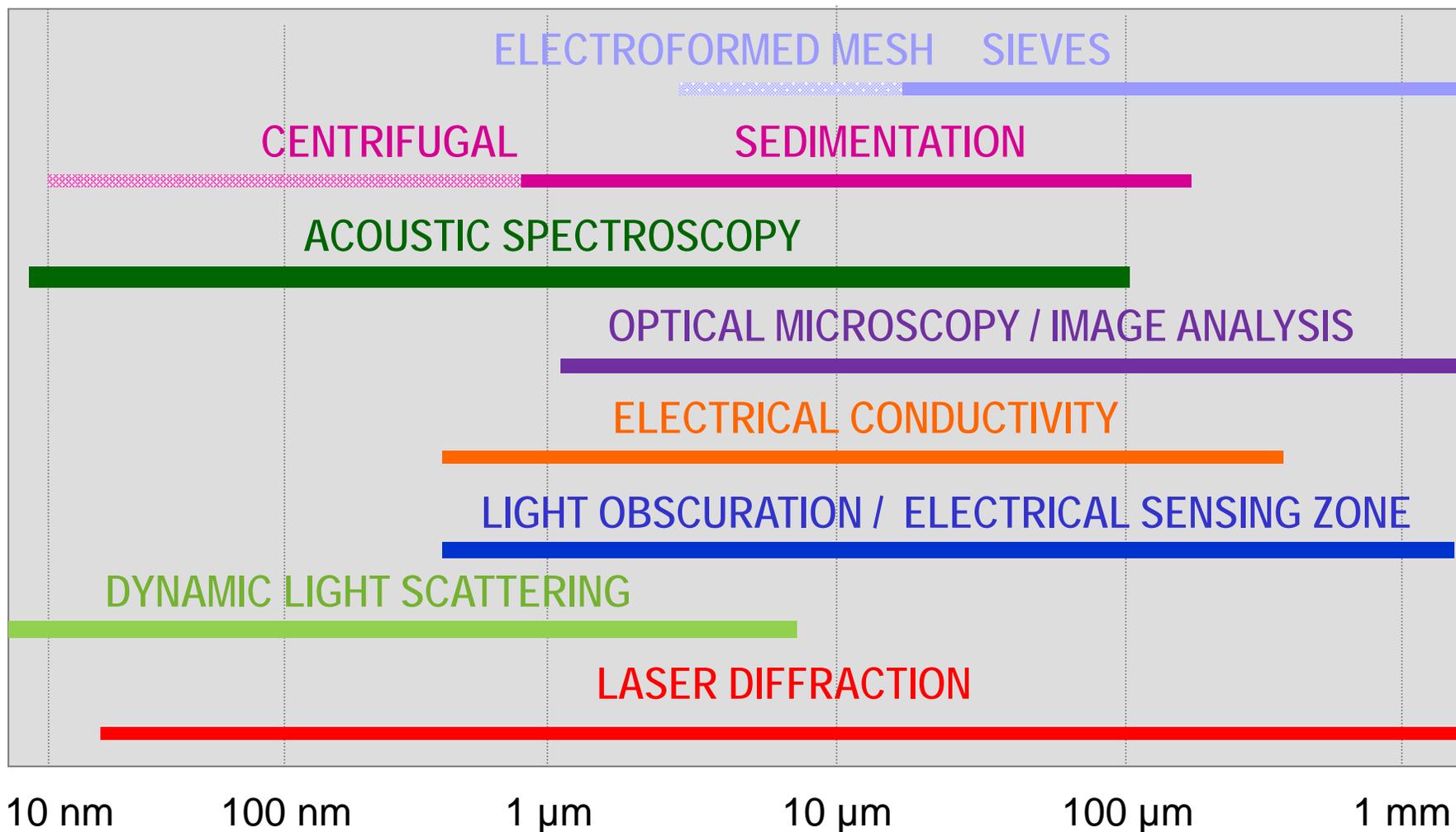
## ■ **PSA300**

Static Image Analysis

## ■ **SA-9600**

Flowing Gas BET Surface Area

# Size Range by Technique



# LA-950: Laser Diffraction



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- Particle size performance leader
- Ninth generation
- Ultra durable
- Lowest total cost of ownership
- Suspension, emulsion, powder, paste, gel
- 10 nanometer – 3 mm



# SZ-100: Dynamic Light Scattering

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- Particle size: 0.3 nm – 8  $\mu\text{m}$
- Zeta potential: -200 – +200 mV
- Molecular weight:  $1 \times 10^3$  –  $2 \times 10^7$  Da
- Patented ultra long-life graphite electrodes
- Lowest total cost of ownership
- Optional autotitrator

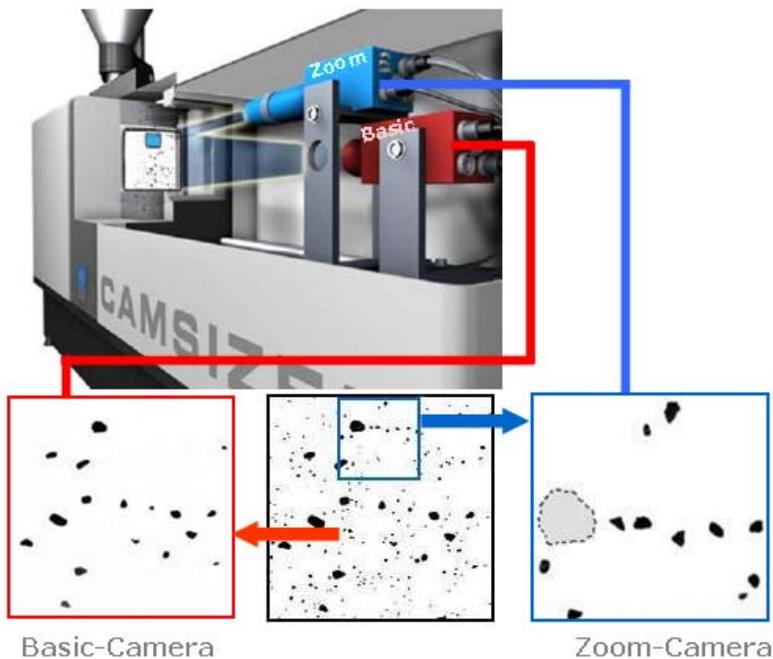


# Image Analysis

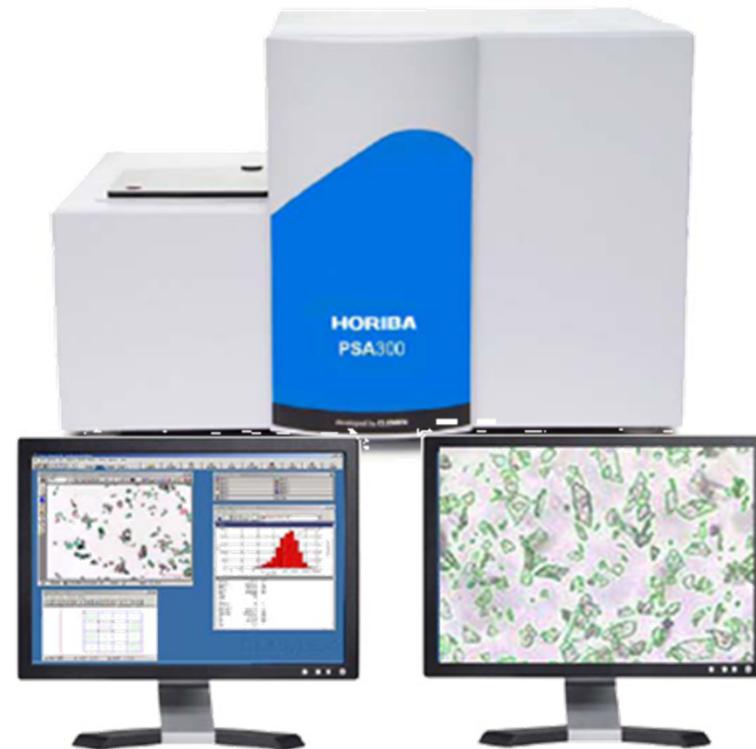


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Dynamic:  
particles flow past camera



Static:  
particles fixed on slide,  
stage moves slide



# CAMSIZER Series



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- High resolution size & shape
- Intelligent sieve correlation
- Patented dual capture
- CAMSIZER
  - 30  $\mu\text{m}$  – 30 mm
  - Free-flowing powders
- CAMSIZER XT
  - 1  $\mu\text{m}$  – 3 mm
  - Cohesive or free flowing



# PSA300



**HORIBA**  
Scientific

- High resolution size & shape
- Referee technique for micronized powders
- Turnkey, automated image analysis
- 1  $\mu\text{m}$  – 1,000  $\mu\text{m}$
- Cohesive or free flowing powders
- Optional Powder Disperser accessory





# What we'll talk about

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- General concerns
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- **Confirming particles < 1 micron**
- Confirming particles > 1 micron
- Q&A

# Likely Technologies

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- **Laser diffraction**
- **Dynamic light scattering**
- **Ultramicroscopy**
- **Electron microscopy**

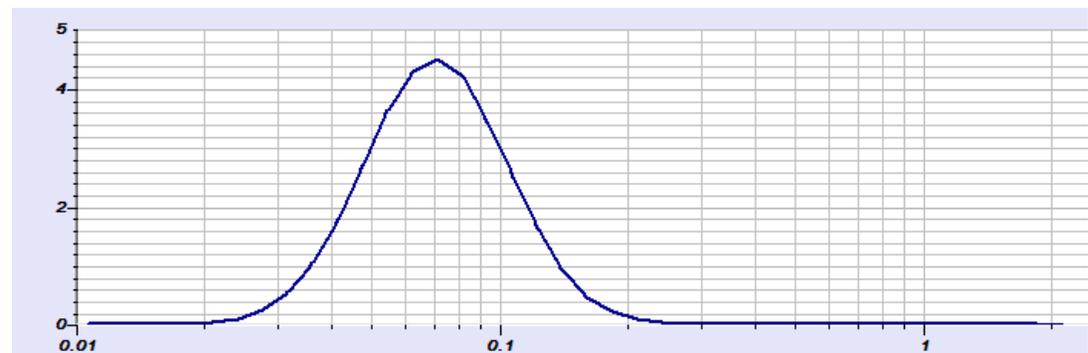
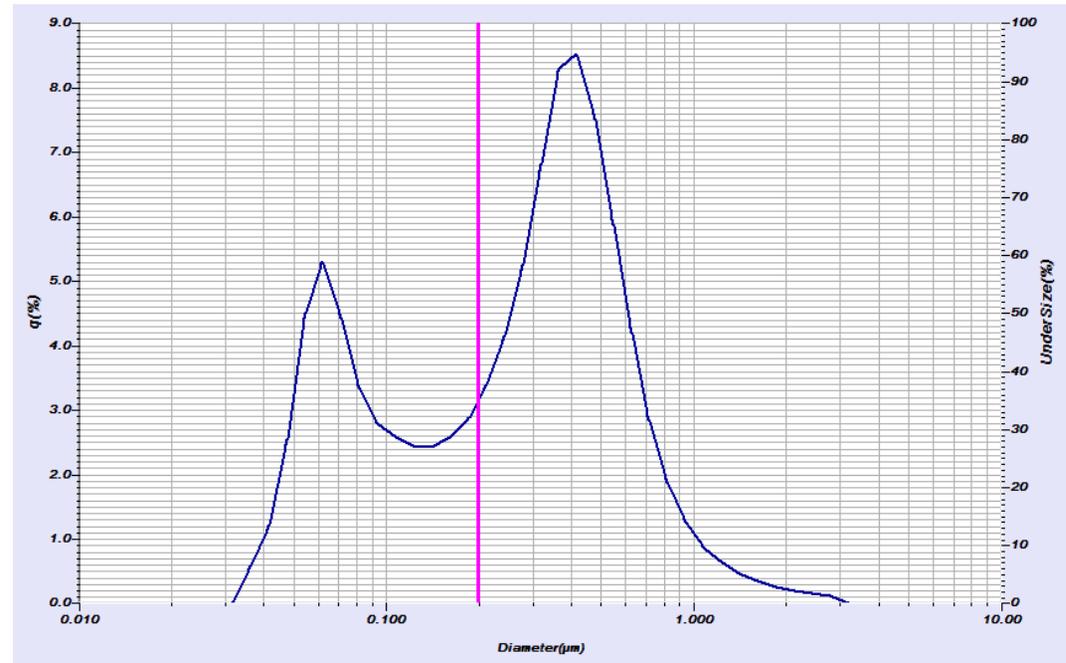
# Laser Diffraction $< 1 \mu\text{m}$



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## ■ Option 1: Filter

- Remove large particles
- Re-measure with laser diffraction and/or DLS
- Reliable measurement?



# Laser Diffraction $< 1 \mu\text{m}$



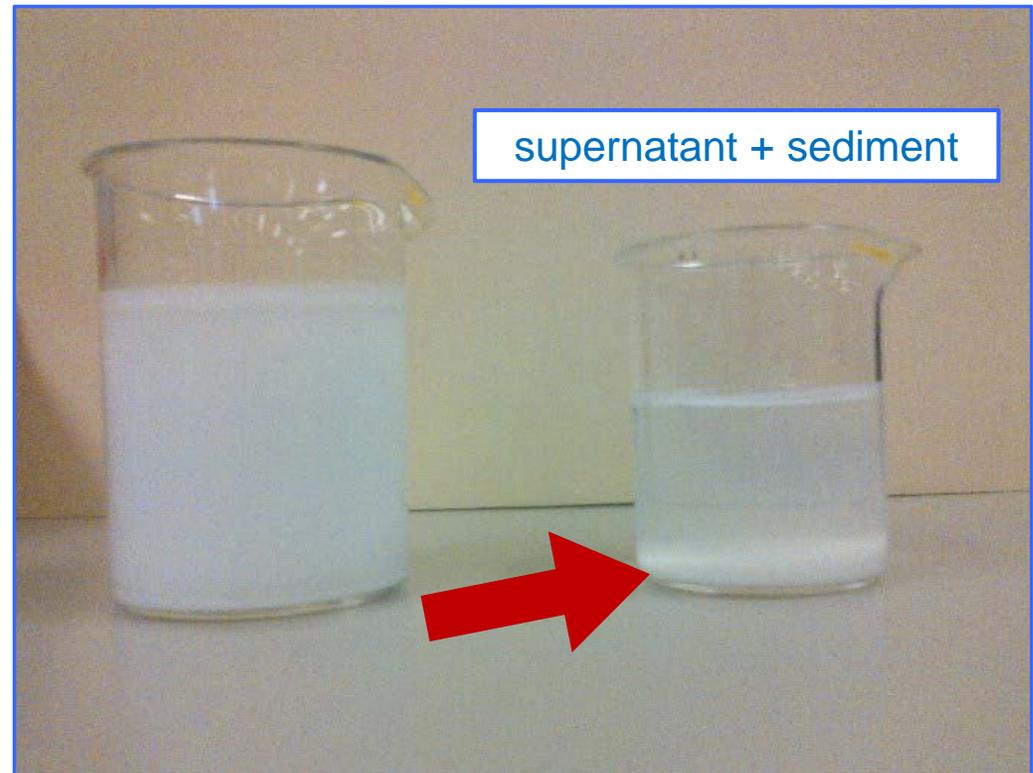
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## ■ Option 2: Sediment

- Create/sample suspension
- Let sit overnight or centrifuge

Small particles will stay suspended by Brownian motion

- *Carefully* sample supernatant
- Re-measure with laser diffraction or DLS
- Reliable measurement?



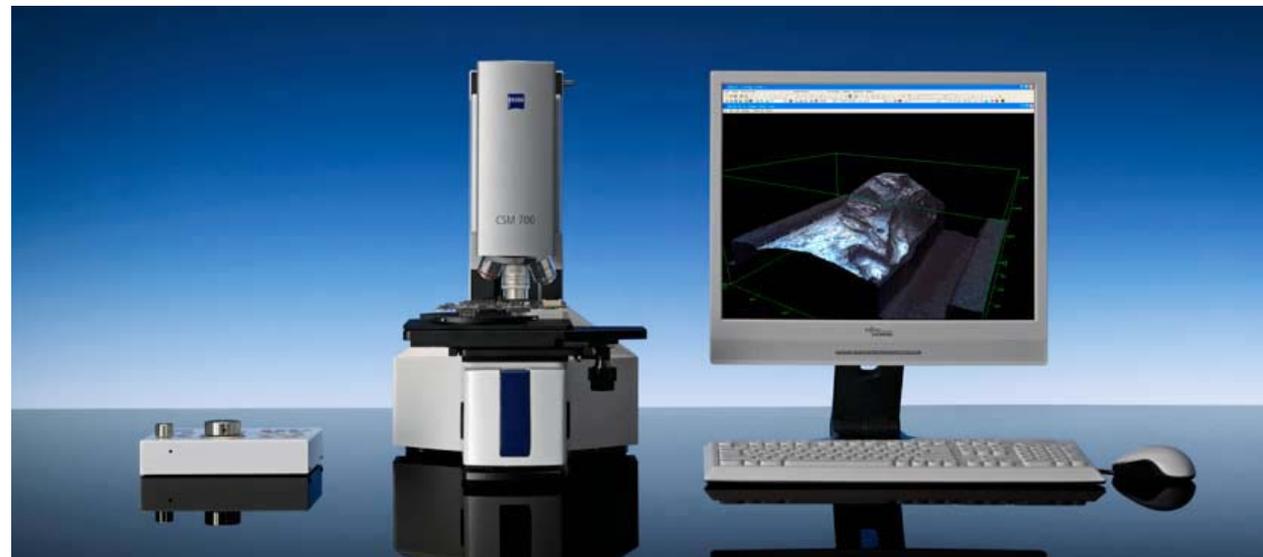
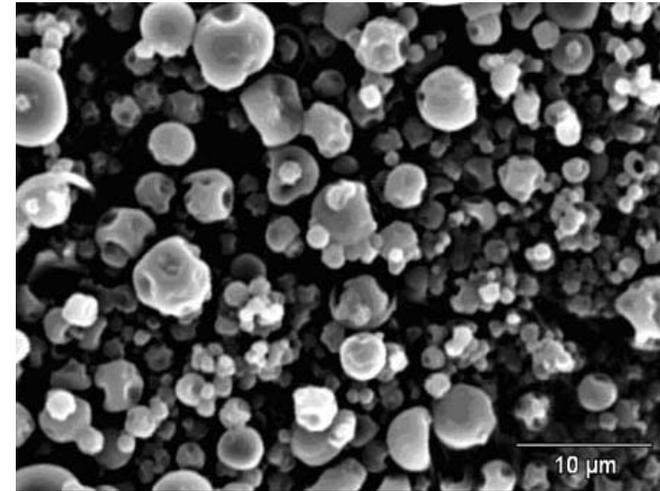
# Laser Diffraction < 1 $\mu\text{m}$



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## ■ Option 3: Electron Microscopy

- Sampling is a concern
- Can confuse primary particles and aggregates
- Very high resolution



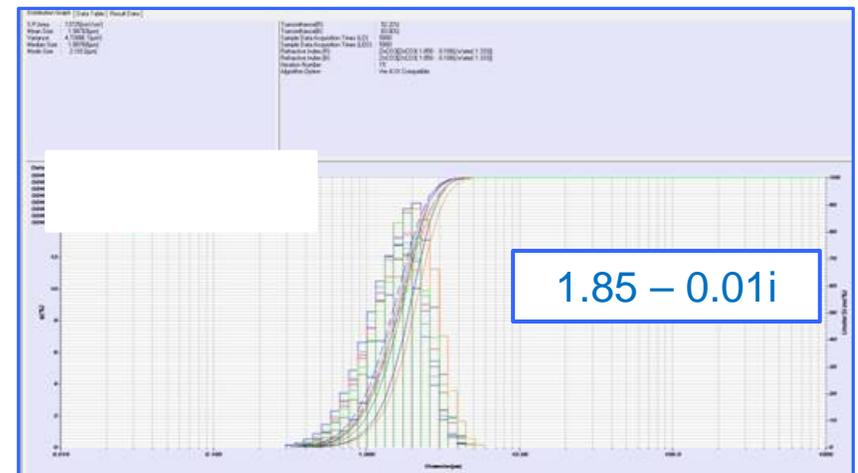
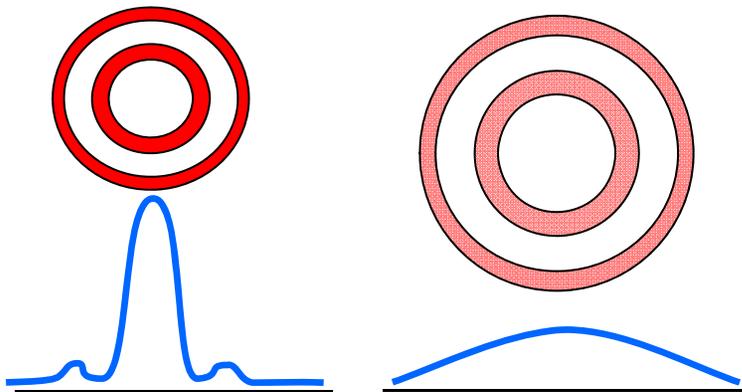
# Laser Diffraction $< 1 \mu\text{m}$

## ■ Option 4: Change RI

- Possible secondary scattering
- Not as definitive as other options
- Use Method Expert software to speed up

Large particles scatter intensely at low angles

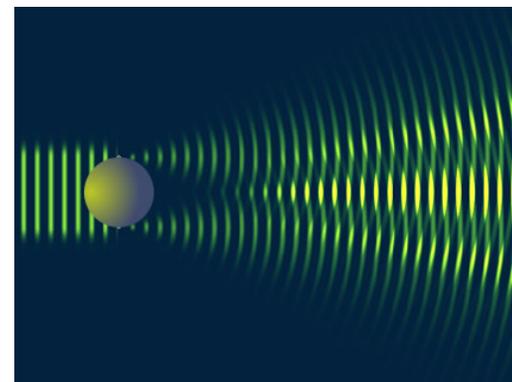
Small particles scatter weakly at high angles



# Dynamic Light Scattering < 1 $\mu\text{m}$

## ■ Option 1: Laser Diffraction

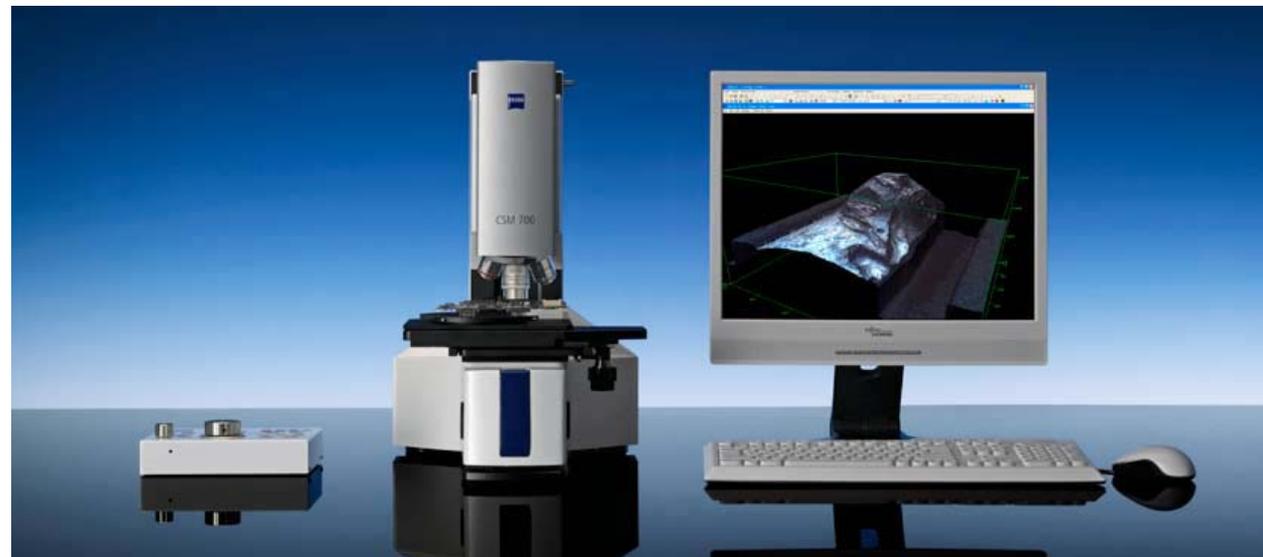
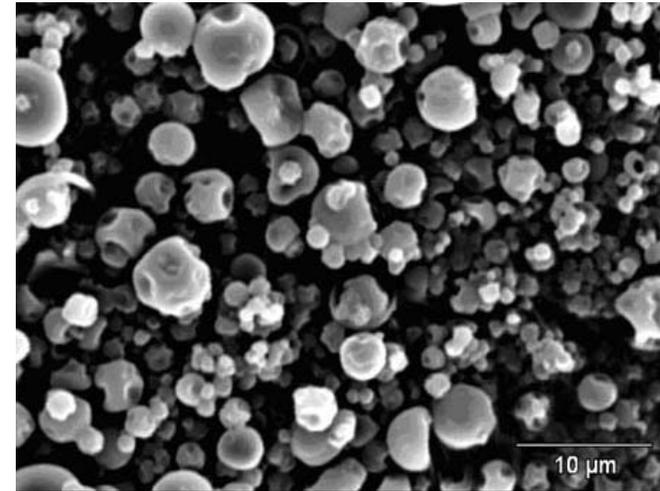
- DLS **very** sensitive to presence of large particles (scattering  $d^6$ )
- Diffraction less sensitive ( $d^3$ ), more likely to see small particles which may be “hidden” by large
- Most advanced diffraction analyzers have detection limit of 10 nm
- Need greater sample quantity than DLS (min. dispersed vol. 5 mL)
- Need greater sample concentration than DLS



# Dynamic Light Scattering < 1 $\mu\text{m}$

## ■ Option 2: Electron Microscopy

- Sampling is a concern
- Can confuse primary particles and aggregates
- Very high resolution



# Dynamic Light Scattering $< 1 \mu\text{m}$

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- Option 3: Filter
  - Remove the large particles to eliminate their scattering





# What we'll talk about

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- General concerns
- Orthogonal techniques
- Confirming particles < 1 micron
- **Confirming particles > 1 micron**
- Q&A

# Likely Technologies

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- Laser diffraction
- Dynamic light scattering
- Image analysis
- Sieves

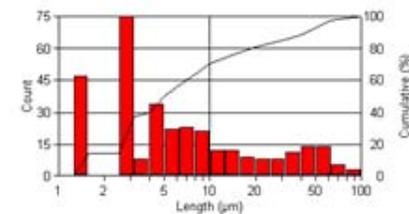
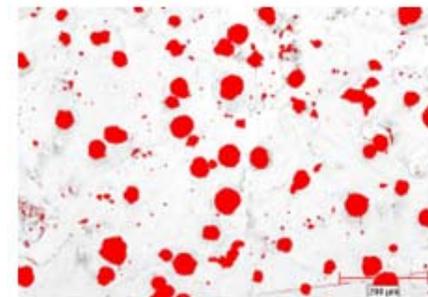
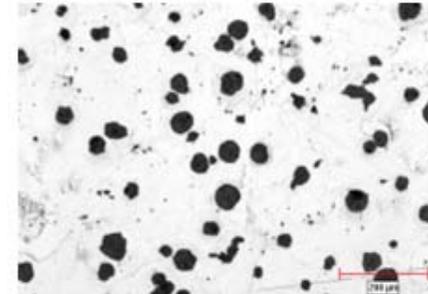
# Laser Diffraction $> 1 \mu\text{m}$



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## ■ Option 1: Static Image Analysis

- Quickly verify large particles
- Provide shape info
- Automated or manual?
- Reliable measurement?



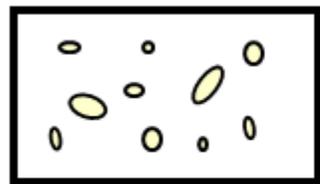
# Laser Diffraction $> 1 \mu\text{m}$



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## ■ Option 2: Sampling & dispersion

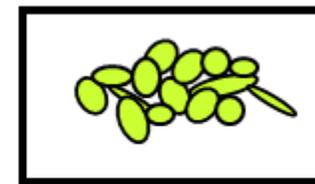
- Large particles are more difficult to sample accurately
- Try different sampling techniques like spin riffling (powders) or magnetic stirring (suspensions)
- Large particles may actually be agglomerates
- Try different surfactants
- Use internal ultrasound
- See webinar TR005 on [horiba.com](http://horiba.com)



**WELL DISPERSED**



**AGGLOMERATED**

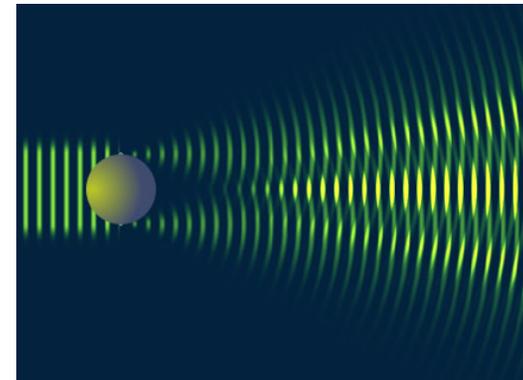


**AGGREGATED**

# Dynamic Light Scattering > 1 $\mu\text{m}$

## ■ Option 1: Laser Diffraction

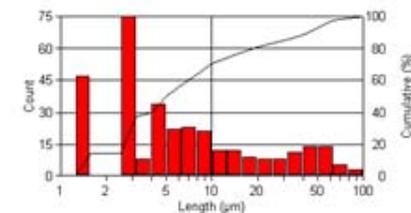
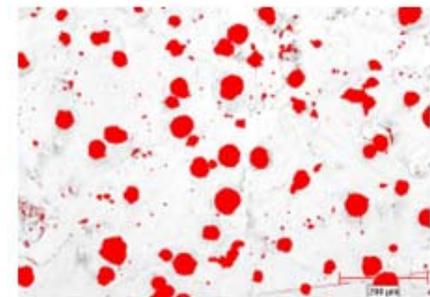
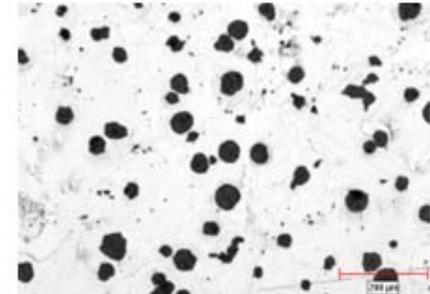
- Not as sensitive to large particles as DLS though doesn't suffer from sedimentation problem
- Most advanced diffraction analyzers have detection limit > 1mm
- Need greater sample quantity than DLS (min. dispersed vol. 5 mL)
- Need greater sample concentration than DLS



# Dynamic Light Scattering $> 1 \mu\text{m}$

## ■ Option 2: Static Image Analysis

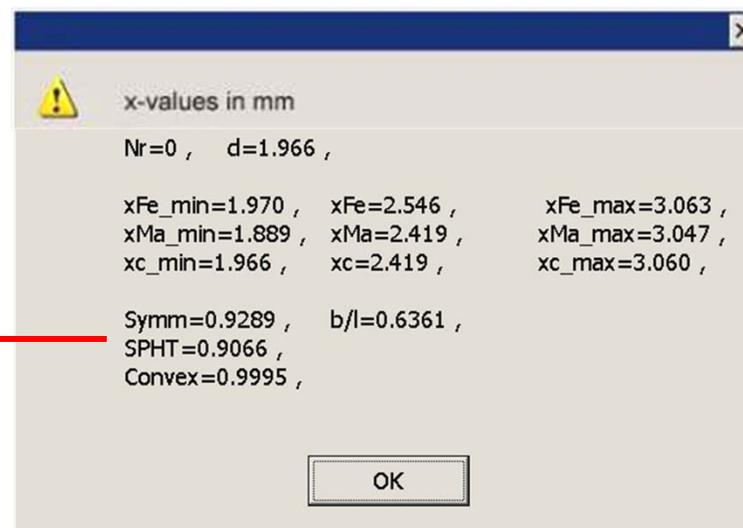
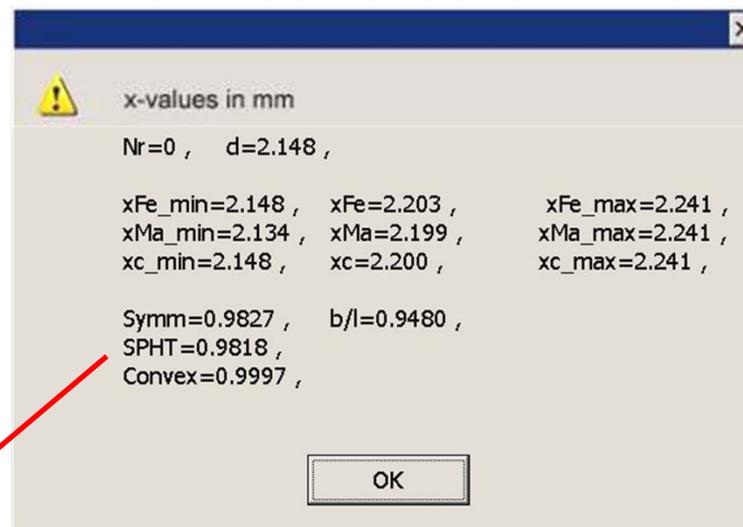
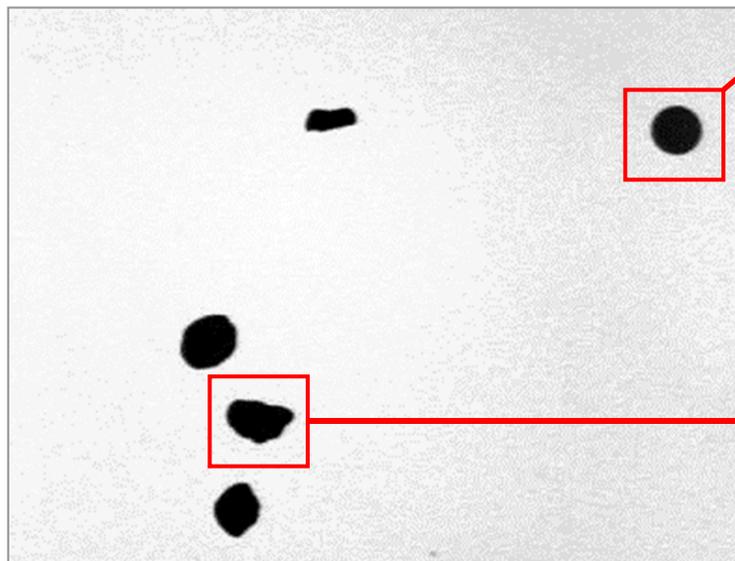
- May have concentration problem for typical DLS applications
- Quickly verify large particles
- Provide shape info
- Automated or manual?
- Reliable measurement?



# Image Analysis > 1 $\mu\text{m}$



- Option 1: Save an image
  - Straightforward, convincing
  - Manual microscopy subjective
  - Automated IA software will save only those particles which meet certain size and shape criteria



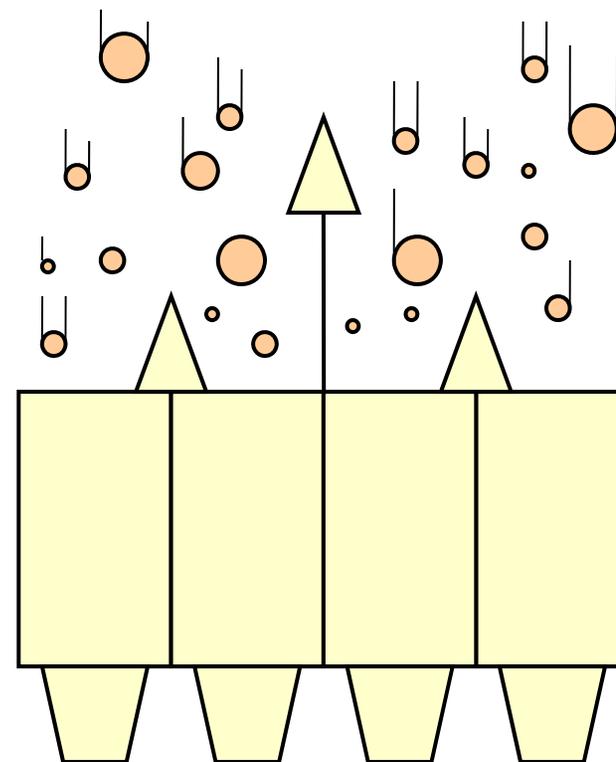
# Image Analysis > 1 $\mu\text{m}$



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## ■ Option 2: Sampling & dispersion

- Large particles are more difficult to sample accurately
- Try different sampling techniques like spin riffling (powders)
- Large particles may actually be agglomerates
- Try different dispersing pressures
- See webinar TR005 on [horiba.com](http://horiba.com)

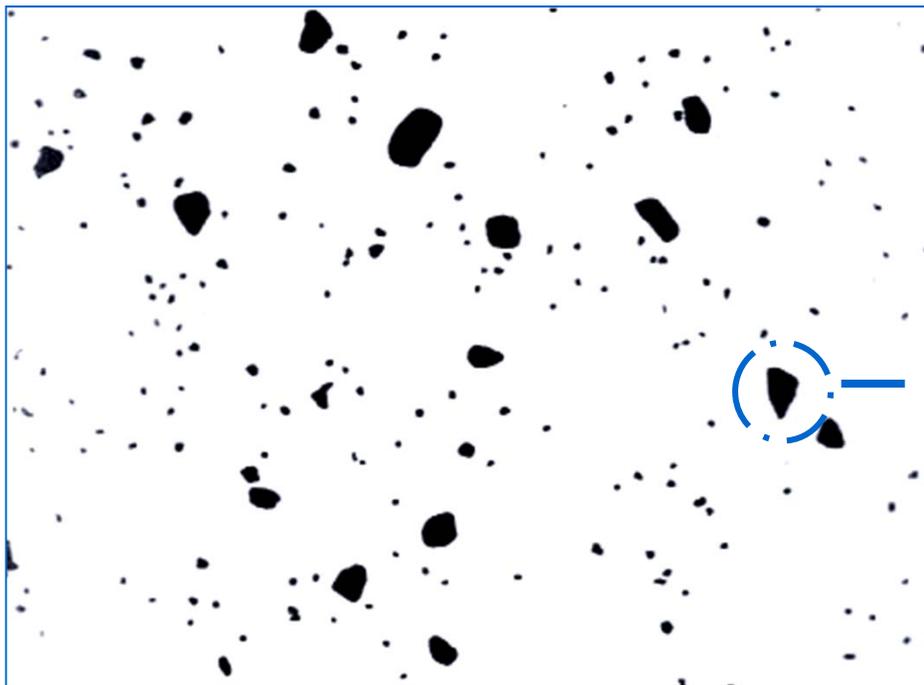


# Expectations

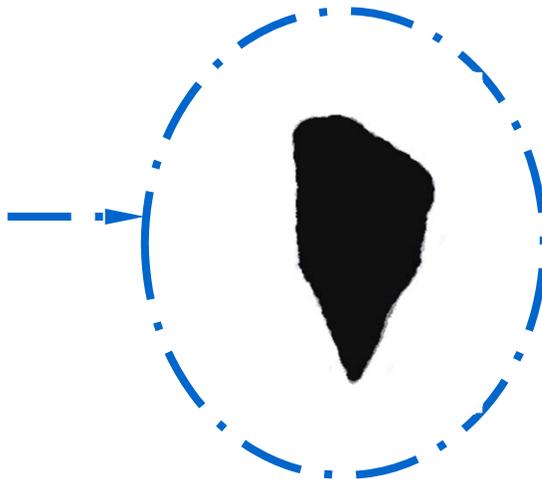


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- What contributed to the size expectation?
- Apples to apples?



What is the size of this particle?



# Equivalent Volume Distributions



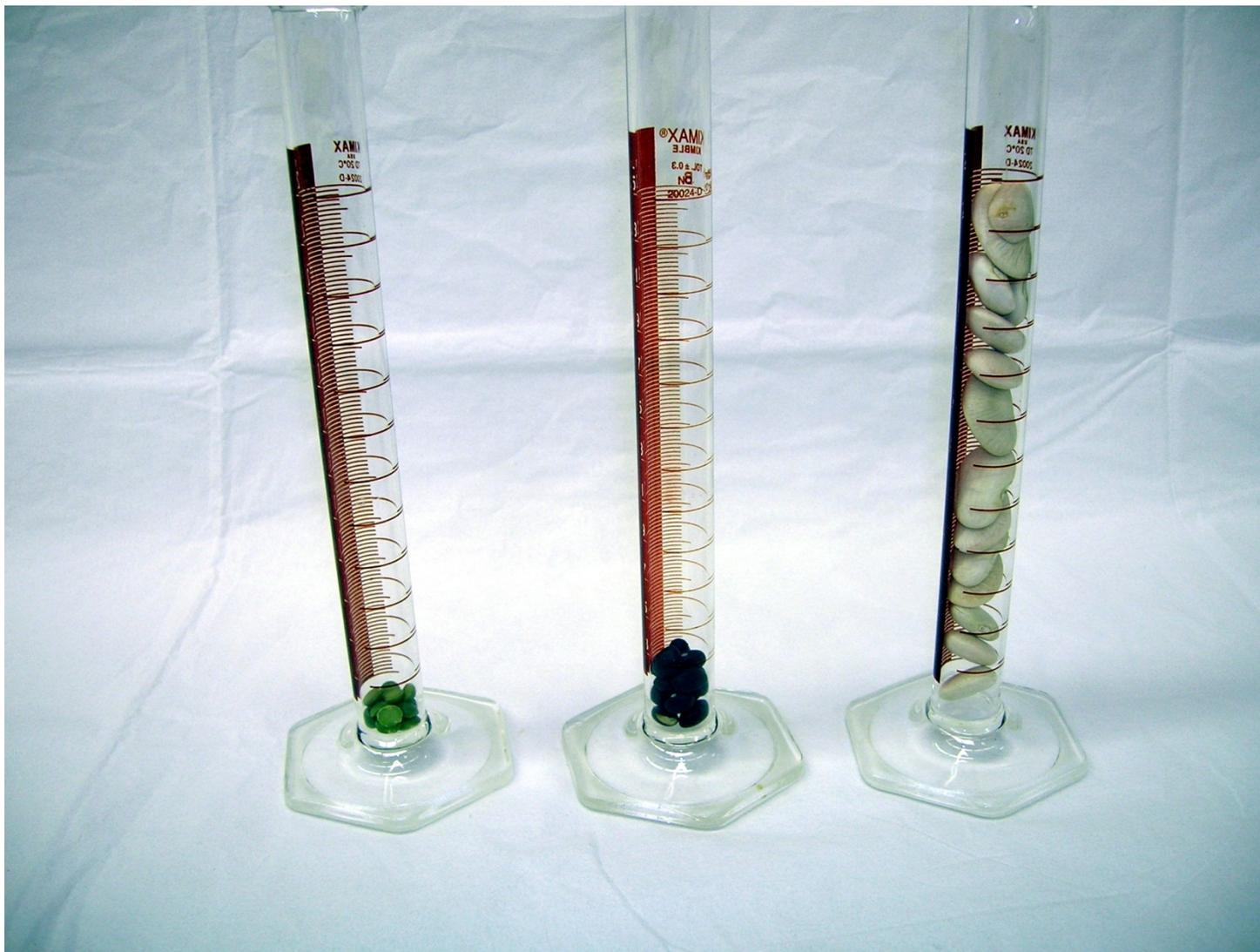
# Equivalent Volume Distributions



# Equivalent Number Distributions

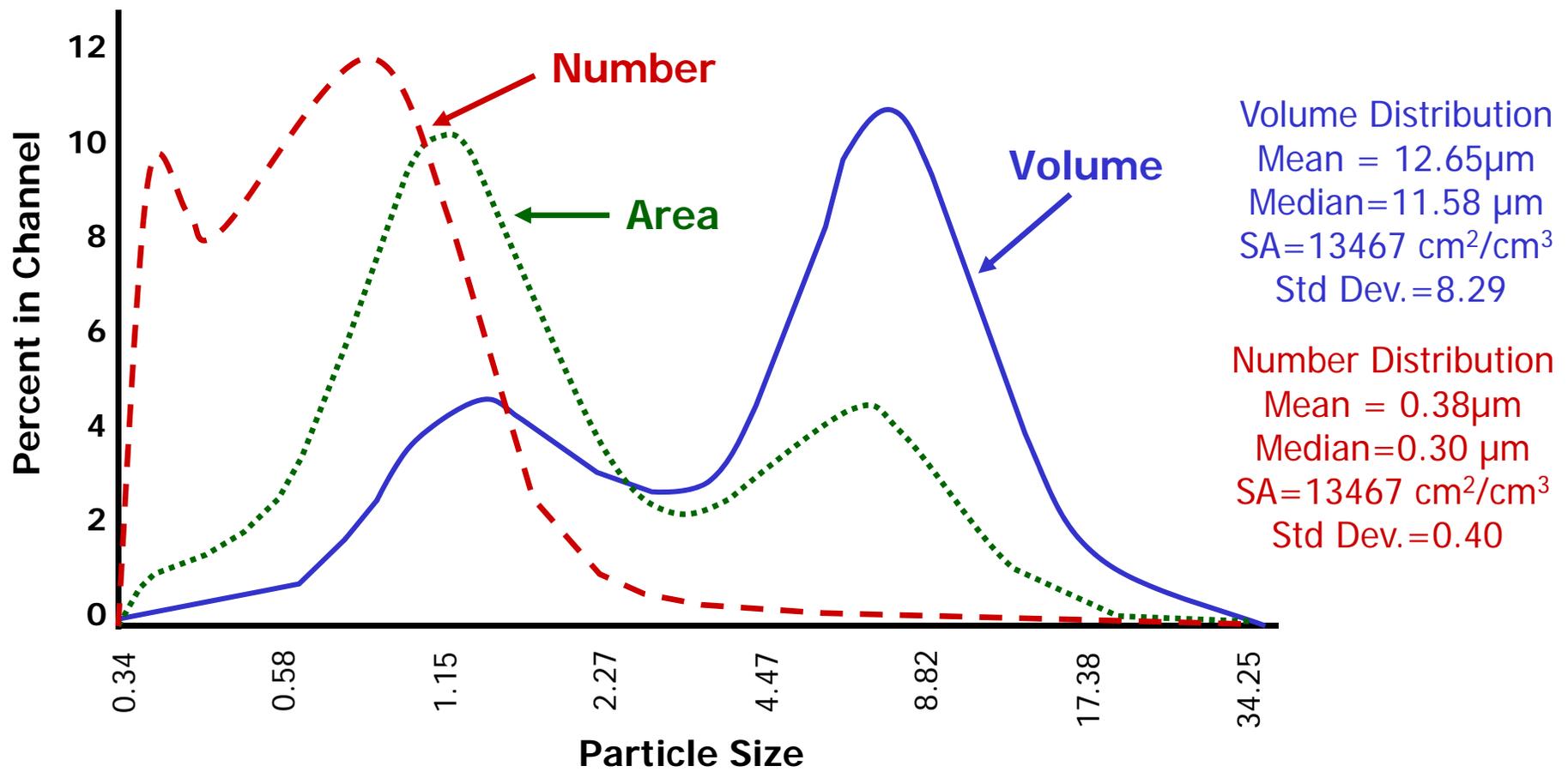


# Equivalent Number Distributions



# Comparing Distribution Bases

- Same material shown as volume, number and area distribution





# Default Bases by Tech

Dynamic light scattering	Scattering intensity
Laser diffraction	Volume
Sieving	Mass (vol. if density constant)
Microscopy (all)	Number
Image analysis	Number
Single particle optical sensing	Number
Electrical sensing zone	Number
Sedimentation (all)	Volume
Acoustic spectroscopy	Volume

# Correlation?

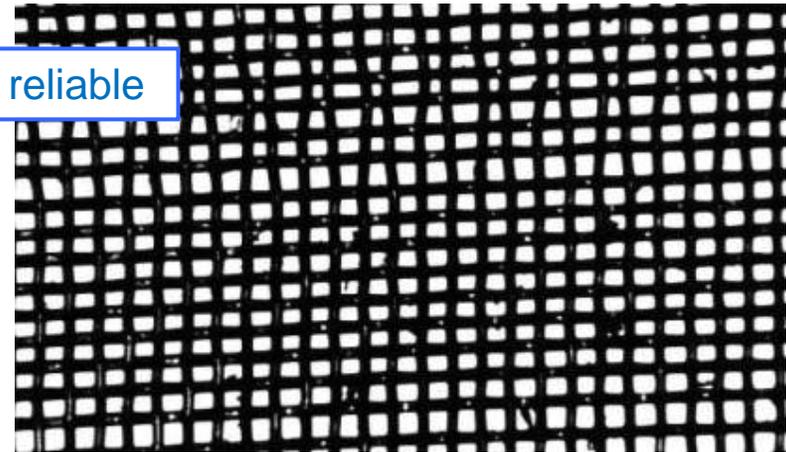


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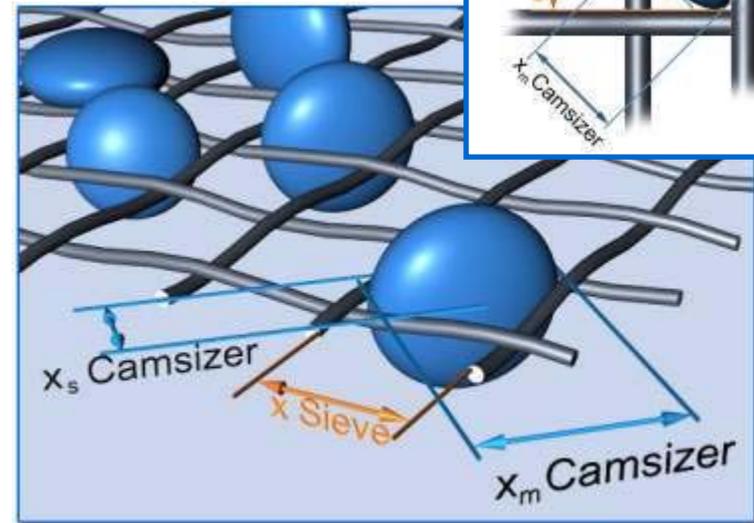
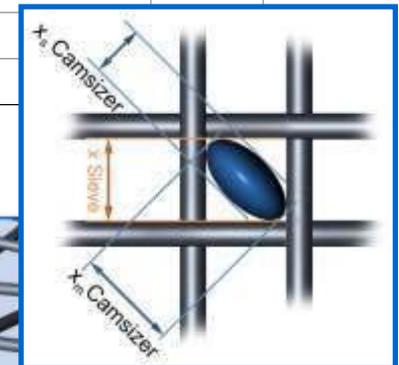
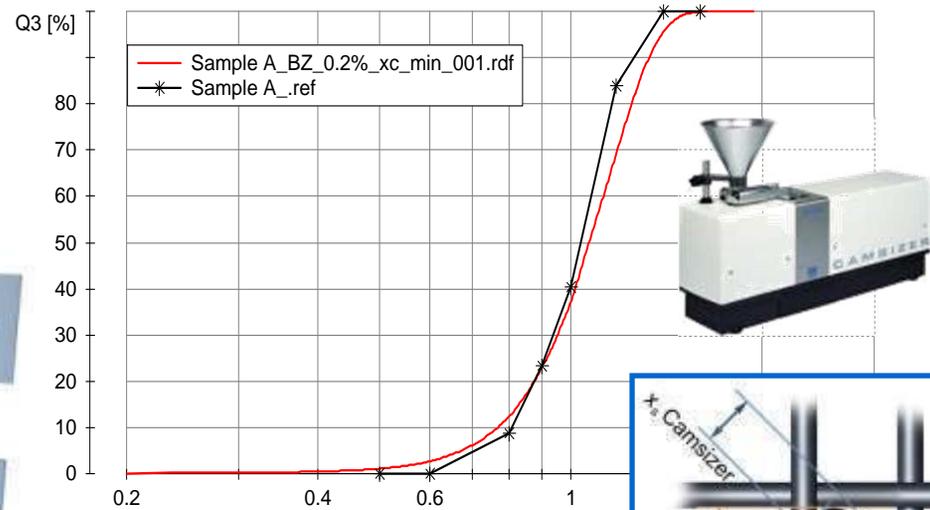
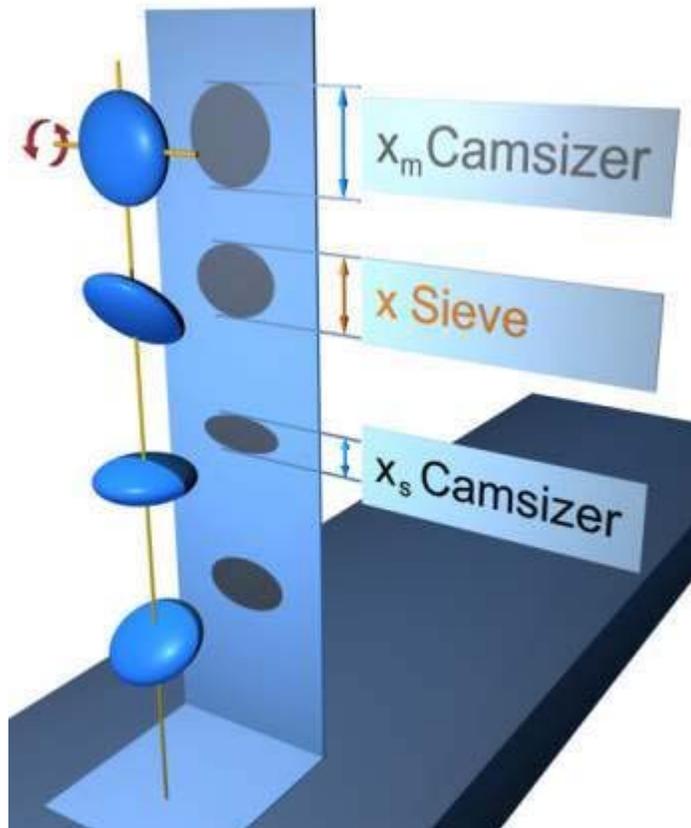
- Make sure data from both techniques is compatible
- Check both tools working correctly

Used sieve mesh, no longer reliable

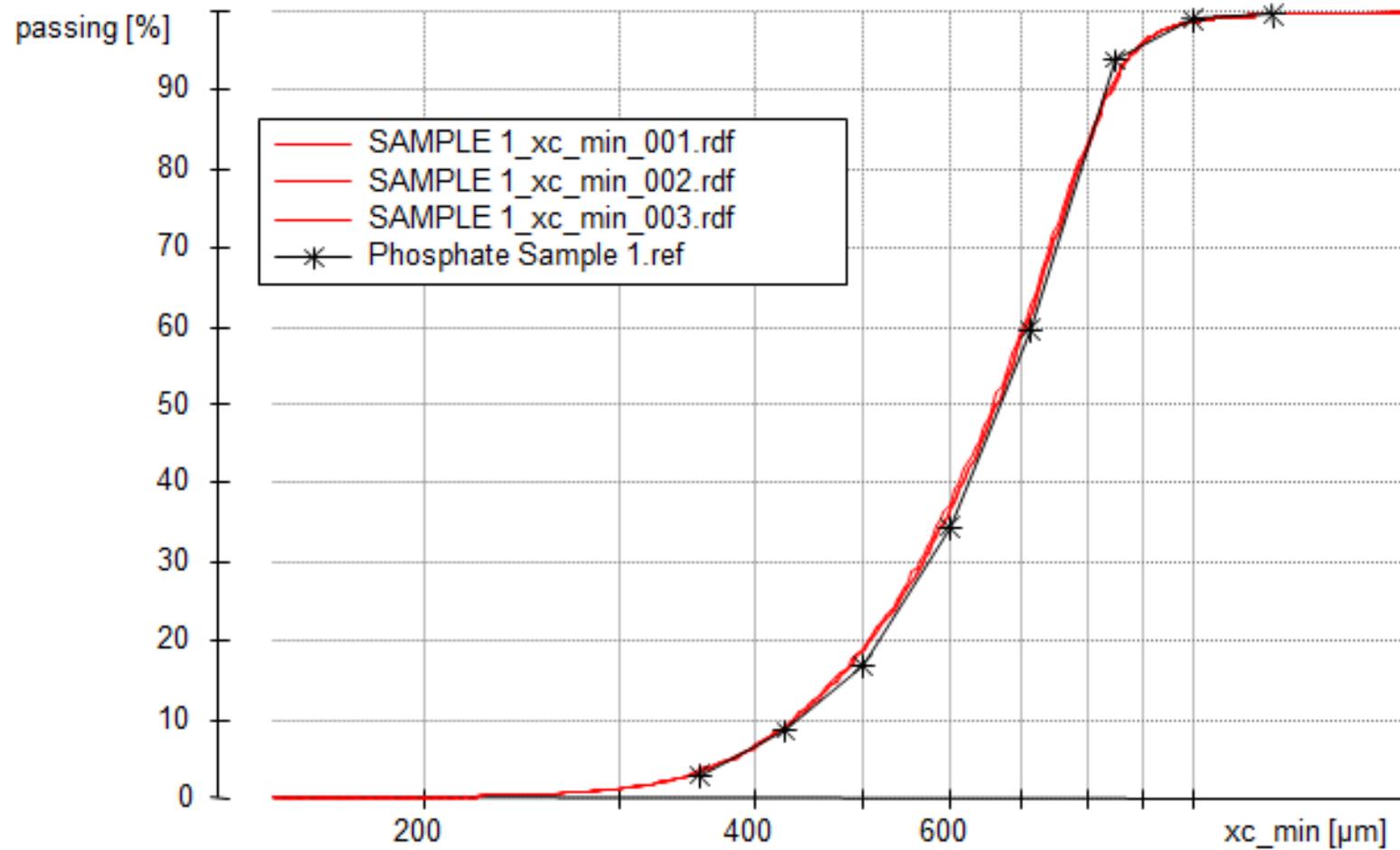
- Is the spec or expectation fair?



# Digitale Imaging ↔ Sieving



# Sieve Correlation





# What we'll talk about

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- General concerns
- Orthogonal techniques
- Confirming particles < 1 micron
- Confirming particles > 1 micron
- **Q&A**



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# Thank you

ありがとうございました

ขอขอบคุณครับ

谢谢

اشكر

Gracias

Grazie

Σας ευχαριστούμε

धन्यवाद

Tacka dig

Danke

Merci

நன்றி

감사합니다

Большое спасибо

Obrigado

おもしろおかしく

*Omoshiro Okashiku*

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Ian Treviranus  
Product Line Manager  
[ian.treviranus@horiba.com](mailto:ian.treviranus@horiba.com)

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## Particle Characterization

Home → Scientific → Products → Particle Characterization

### Particle Characterization

HORIBA designs, manufactures, and supplies state of the art particle characterization instruments.

Every instrument across the five business segments must meet stringent requirements before the HORIBA name is attached. The Particle Characterization group of analyzers has incorporated this principle into each new design since entering the business in 1979. Relentless innovation united with high performance to attain the ultimate goal: a new standard in usability.

#### Particle Characterization Products

HORIBA offers instruments for particle size, particle shape, zeta potential, and surface area analysis. Measurable particle size range is from 1 nanometer to 30 millimeters, at concentrations ranging from 1 ppm to 50 vol% with shape determination available starting at 1 micrometer. A range of analytical techniques are employed including laser diffraction (Mie Theory), dynamic light scattering, acoustic and electroacoustic spectroscopy, and dynamic and static image analysis. (measuring both particle size and shape information).

HORIBA's advanced designs and powerful software, combined with flexible sample handling systems are available to meet every analysis need. These instruments can incorporate small volume pumping systems for precious materials, high throughput automation, dry powder dispersers and temperature controlled flow systems in order to provide the user with the best possible solution with none of the trade-offs that might otherwise be necessary.

#### Particle Size

- Laser diffraction
  - LA-950V2
  - LA-300
- Dynamic light scattering
  - SZ-100

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**Particle Size Essentials**

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