



# Troubleshooting Laser Diffraction Particle Size Results



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# What we'll talk about

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- **Typical workflow**
- Calculation optimization
- Hardware optimization
- LA-950/960 data analysis tools

# Additional resources

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- Number vs. volume (TR001)
- CMP slurry application (AN179)
- Data interpretation (larger topic, TR008)
- Dynamic light scattering (TE012, TR012, TR014)



# Typical workflow

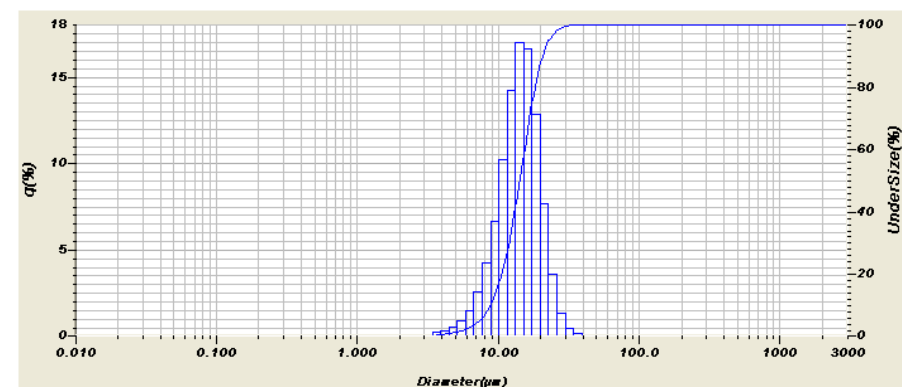
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- Run standard material (TR004)
  - PASS → Proceed
  - FAIL → Check SOP, contact HORIBA
- Compatible technologies?
  - YES → Compare Conditions
  - NO → How are they different
- Compare Conditions
  - SAME → Investigate scattering pattern
  - DIFFERENT → Are differences relevant for this material?
    - YES → Re-test with new conditions
    - NO → Investigate scattering pattern
- When you hit a wall → Ask the experts!  
[labinfo@horiba.com](mailto:labinfo@horiba.com)

# Run standard material

- NIST-traceable or internal reference
- Polydisperse preferable to monodisperse
- Measure at least 3 repeats
- System verification webinar (TR004)

PS202 (3-30μm)	D10	D50	D90
Standard Value (μm)	9.14	13.43	20.34
Uncertainty (μm)	0.86	0.86	1.44
ISO standard error	5%	3%	5%
Lower limit (μm)	7.866	12.193	17.955
Measured Result (μm)	9.721	13.916	18.959
Upper Limit (μm)	10.500	14.719	22.869



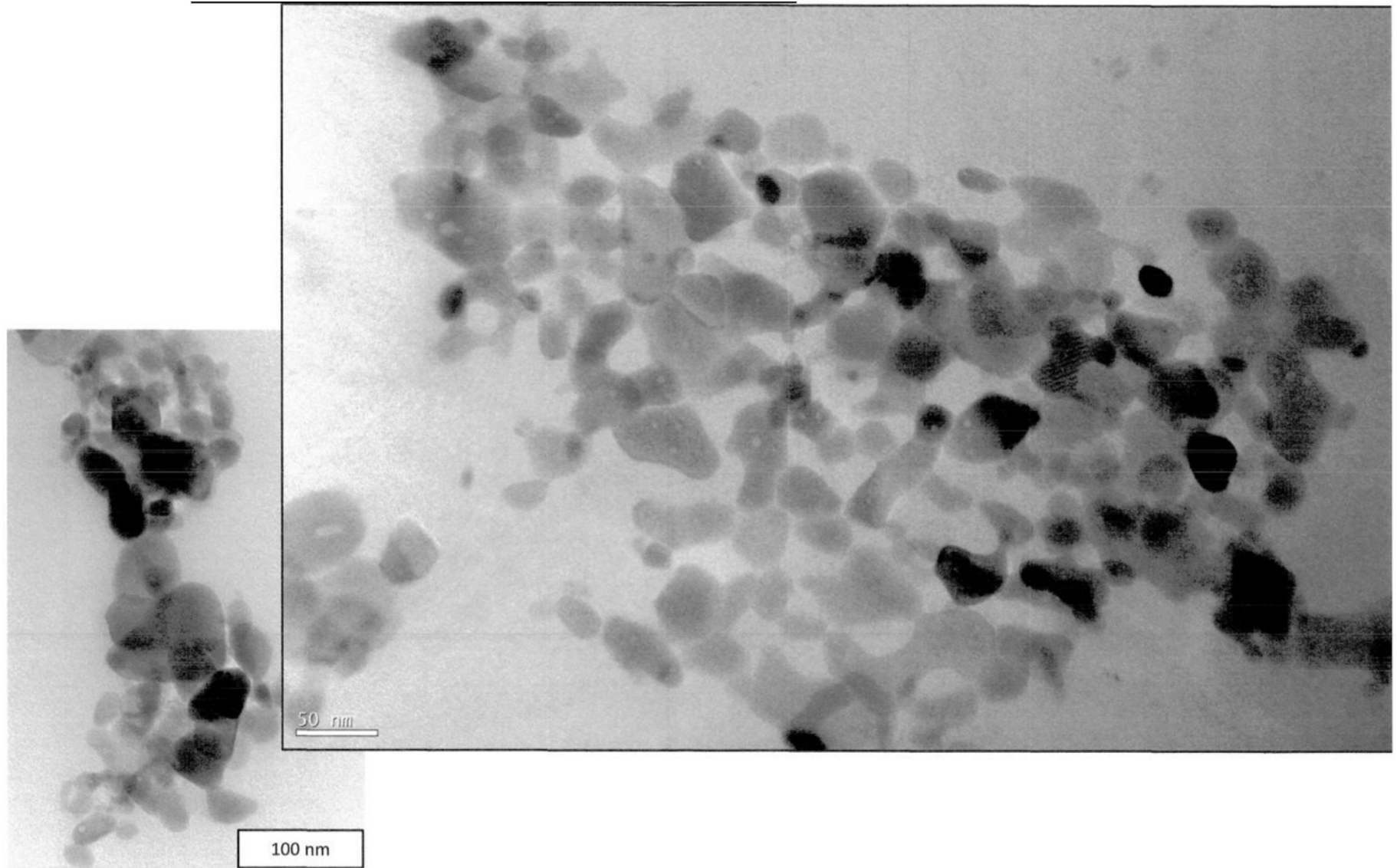
# Compatible technologies

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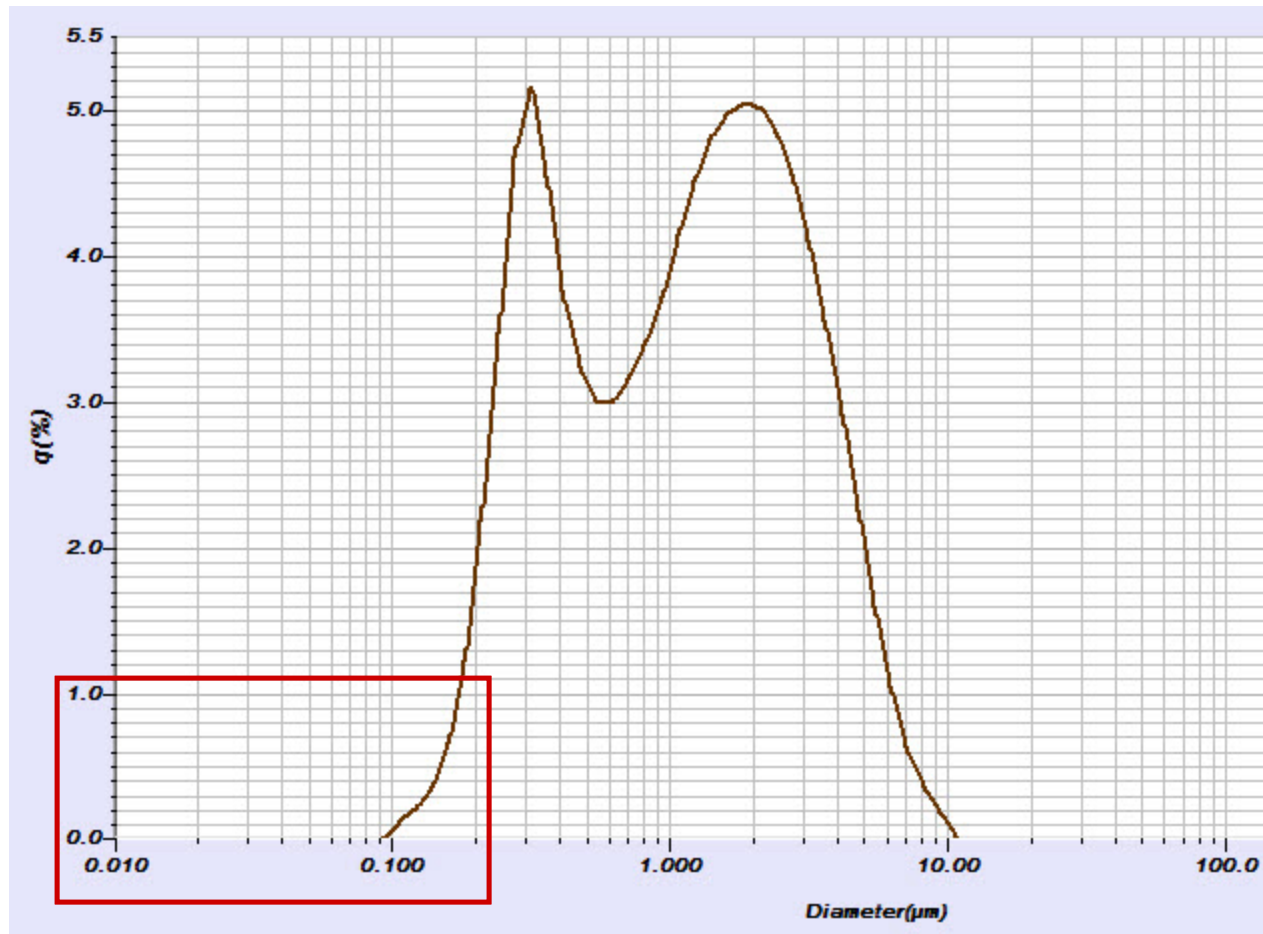
- Different technologies measure different material properties
  - Ideally the comparison is apples to apples
    - Different instruments of same technology are close enough (Gala to Fuji)
  - Diffraction to SEM (or similar) is like apple to oranges and must be approached differently
    - Different technologies gives you more information, not necessarily bad



# Quick example



# LA-960 result



**SEM data alone makes us think this is the size range**



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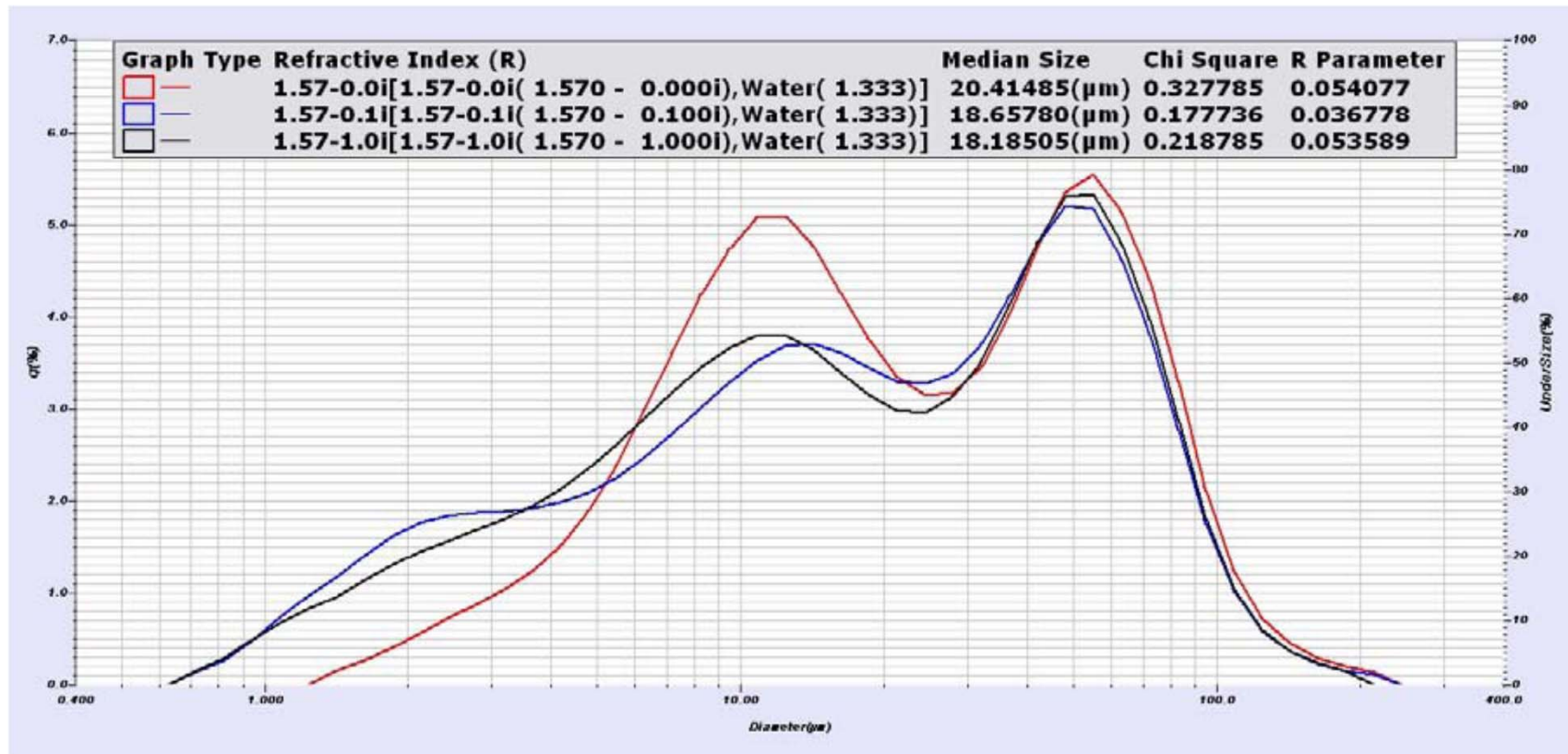
# Compare conditions

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- First, check calculation conditions
  - Refractive Index: real and imaginary, RRI?
  - Distribution Base: volume is best
  - Iterations: wide or narrow size range
- Second, check hardware conditions
  - Concentration: transmittance
  - Particle support: pumping
  - Dispersion: US for wet, air pressure for dry
  - Duration: wide distributions

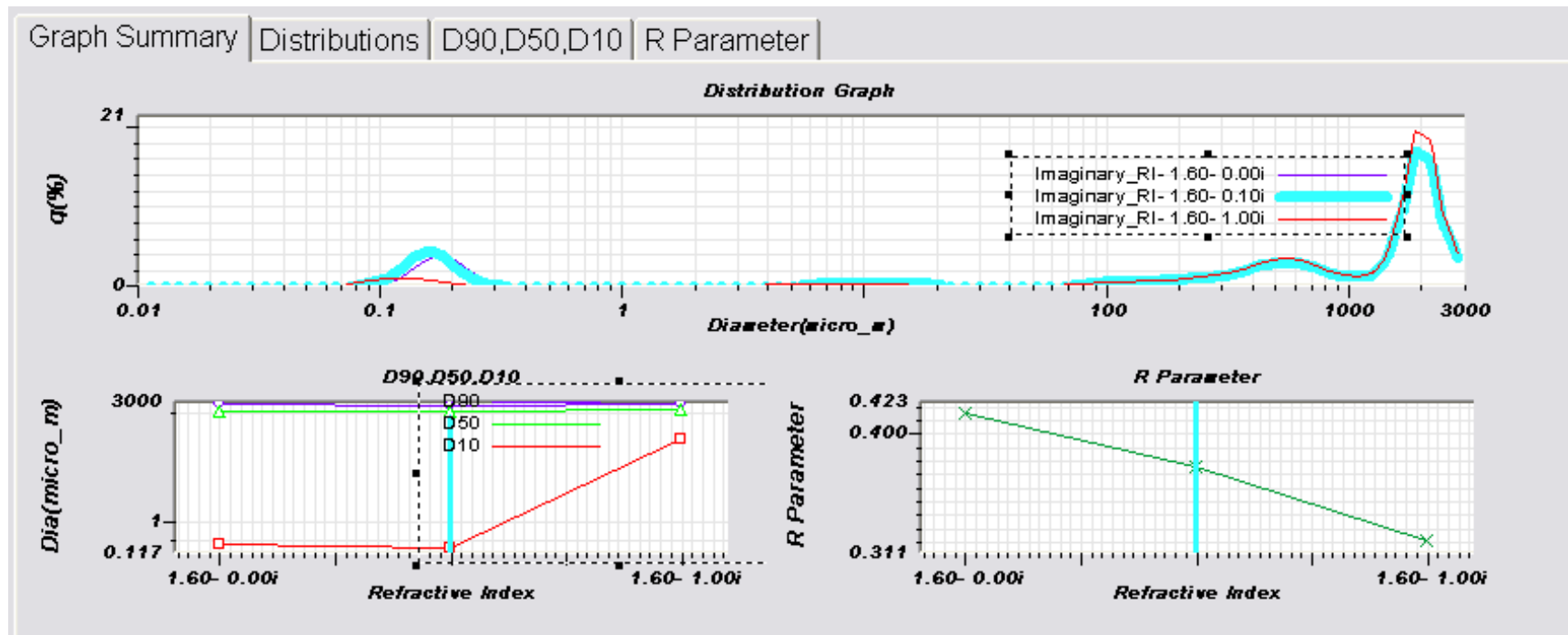
# Refractive index

- Seemingly minor differences calculate different distributions



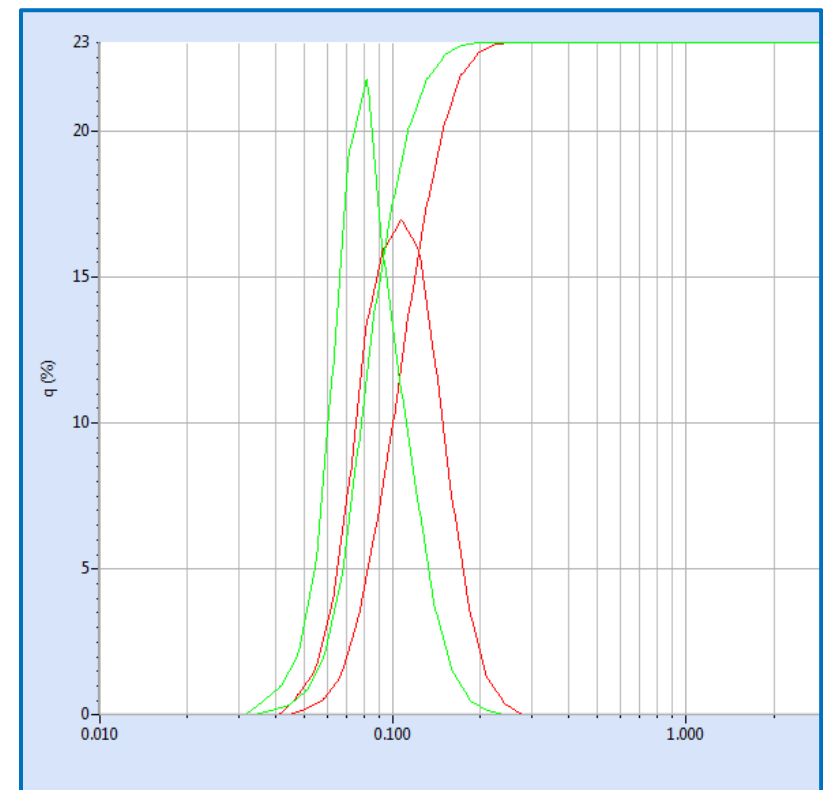
# Refractive index

- Compare easily with LA-960 Method Expert software



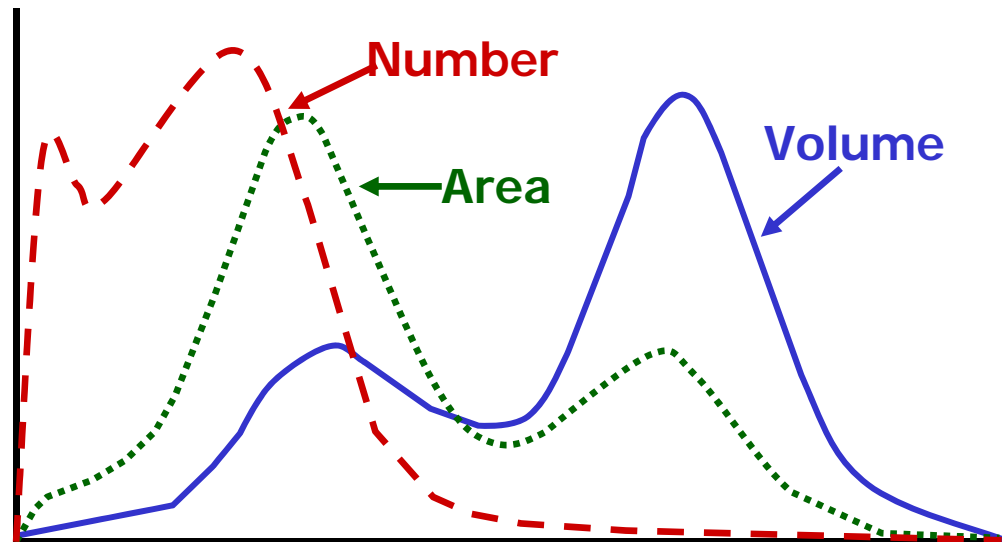
# Refractive index

- RI or RRI?
- Red result is 1.45-0.0i in water (1.33)
- Green result is 1.09-0.0i ... which multiplied by 1.33 is 1.45!



# Distribution base

- Volume basis by default
  - Excellent for mass balancing
  - Number basis recalc → significant error



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# Compare conditions

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## ■ First, check calculation conditions

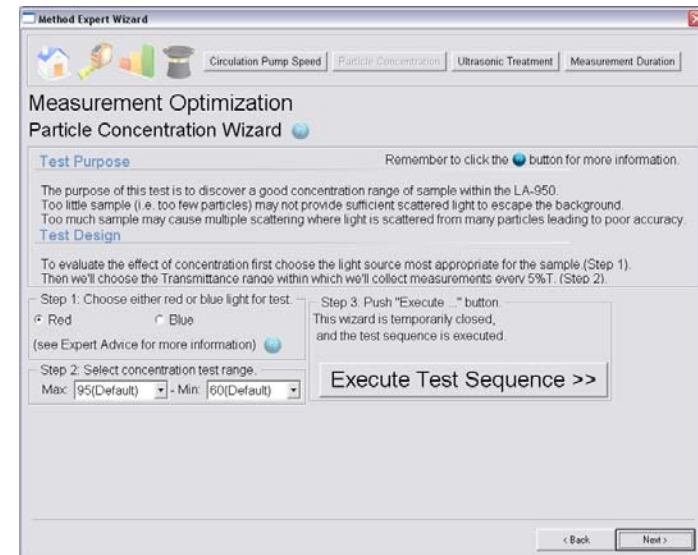
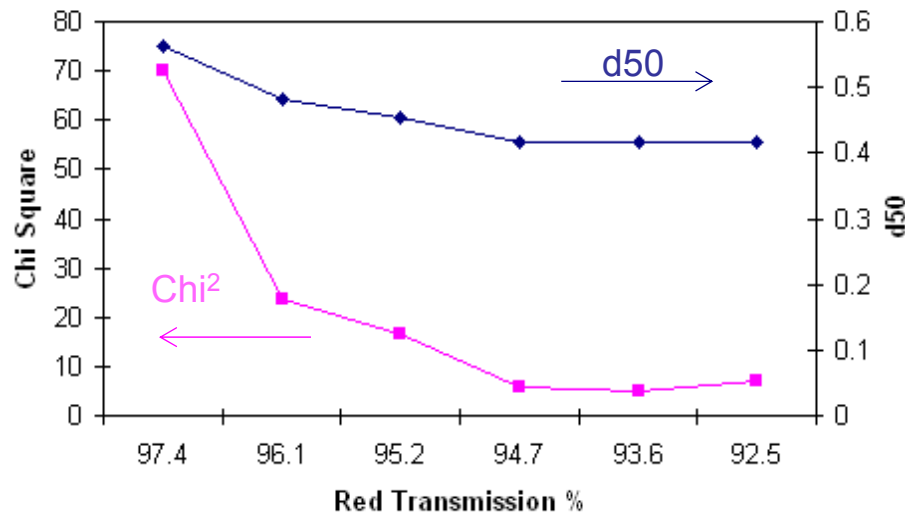
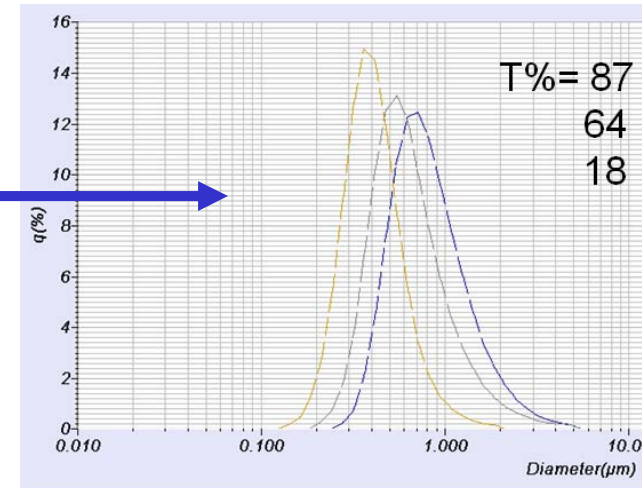
- Refractive Index: real and imaginary
- Distribution Base: volume is best
- Iterations

## ■ Second, check hardware conditions

- Concentration: transmittance
- Particle support: pumping
- Dispersion: US for wet, air pressure for dry
- Duration: wide distributions

# Concentration

- High enough for good S/N ratio
- Low enough to avoid multiple scattering
- Typically 95 – 80 %T
- Measure at different T%, look at Chi Square calculation



# Pump & stirrer

- Must be high enough to suspend & circulate heavy particles
- Not so high that bubbles are introduced
- Adding energy – can disperse loose agglomerates
- Measure at several settings & select optimum
- Can be automated in software (see right)

Exp #	Agitation	Circulation	D <sub>mean</sub> (nm)	D <sub>10</sub> (nm)	D <sub>90</sub> (nm)
1	1	1	187.03	137.5	245.7
2	1	3	184.23	135.9	242.1
3	3	1	187.28	137.8	245.8
4	3	3	184.61	136.1	242.5
5	1	1	185.32	136.3	243.7
6	1	3	184.04	135.8	241.8
7	3	1	184.13	135.8	241.9
8	3	3	184.98	136.4	242.9
Parameters Selected: Agitation: <u>2</u> Circulation: <u>2</u>					

## Automation Wizard

### Measurement Preparation

Step 1: How will dispersant be delivered to the instrument? How much?

Choose fill level  Wait time after  (s)

Step 2: Choose a circulation pump speed

Choose pump speed  Wait time after  (s)

Step 3: Choose a agitation stirrer speed.

Choose agitator speed  Wait time after  (s)

Step 4: Debubble the instrument?

☐ No ☐ Yes Wait time after  (s)

Step 5: Blank check of instrument cleanliness?

☐ No ☐ Yes Wait time after  (s)

< Back Next >

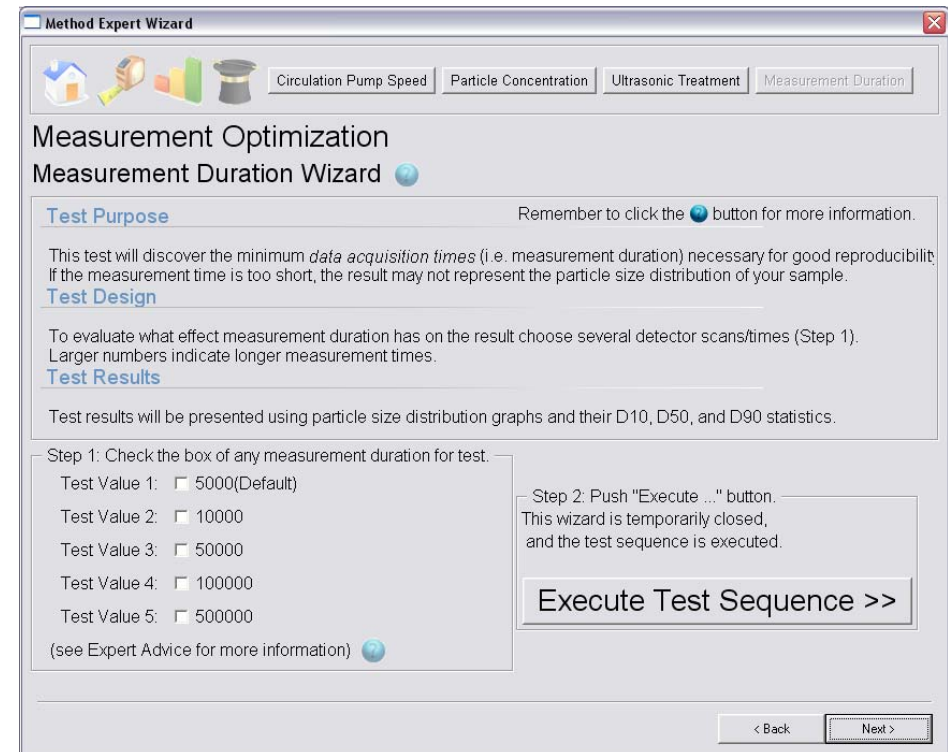
# Ultrasonic dispersion

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- Adding energy to break up agglomerates – disperse to primary particles, without breaking particles
- Similar to changing air pressure on dry powder feeder
- Typically set to 100% energy, vary time (sec) on
- Investigate tails of distribution
  - High end to see if agglomerates removed
  - Small end to see if new, smaller particles appear (breakage)
- Test reproducibility, consider robustness
- Note:
  - Can break emulsions (or have no effect)
  - Can cause thermal mixing trouble w/solvents - wait
  - Use external probe if  $t > 2-5$  minutes

# Measurement duration

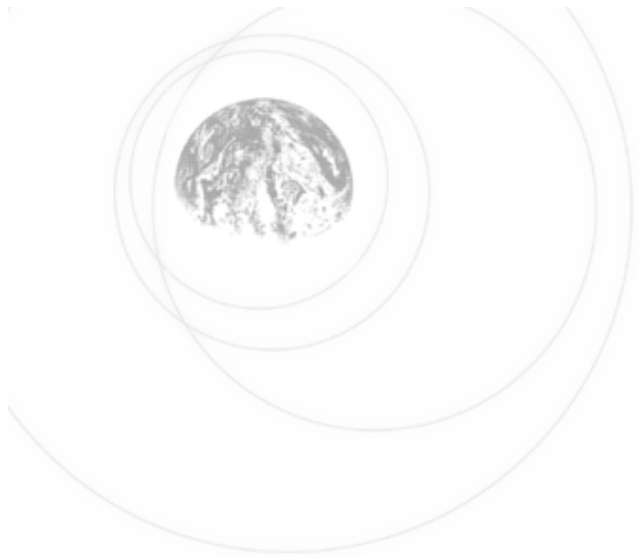
- Long enough for reproducibility
- Typically 5 sec, up to several minutes
- Longer time for large, broad distributions
- Can be automated in software



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# Choosing Parameters



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# Choosing good statistics

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## Statistics describing the distribution must...

Tell us about our process

Be relevant

Be controlled well

**Be reproducible!**

## Poor precision is the result of either a poor method or poor statistical choices

**We can help!**  
**[labinfo@horiba.com](mailto:labinfo@horiba.com)**

# The basis for reliable data

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## Reproducibility!

Prepare, measure, empty, repeat

## What would be good reproducibility?

Look at the accepted standards

### ISO 13320

COV < 3% at Median (D50)

COV < 5% at D10 and D90

### USP <429>

COV < 10% at Median (D50)

COV < 15% at D10 and D90

$$\text{COV} = 100 * (\text{StDev} / \text{Mean})$$

Note: All limits double when  $D50 < 10 \mu\text{m}$

Note: Must acquire at least 3 measurements from unique samplings

# Calculation automation

**Select Summary Items**

Item List  
Test or Assay Number  
Remarks 1  
Remarks 2  
Remarks 3  
Remarks 4  
Remarks 5  
Remarks 6  
Remarks 7  
Remarks 8  
Remarks 9  
Remarks 10

Add >>  
Delete

Summary Items  
Sample Name  
Material  
Source  
Lot Number  
D(v,0.1)  
D(v,0.5)  
D(v,0.9)

Clear Up Down

Font: MS Sans Serif Font Open

Orientation: ☒ Portrait ☐ Landscape

☒ Show Summary Averages ☒ Show Summary Std. Dev.  
☒ Show Coefficient of variation(Relative Std. Dev.)

Validation:  
Specification: USP 429

D(v,0.1) Range (± %): 15 10 15  
D(v,0.5) Range (± %): 30 20 30

Save As Cancel OK

**Summary Report**

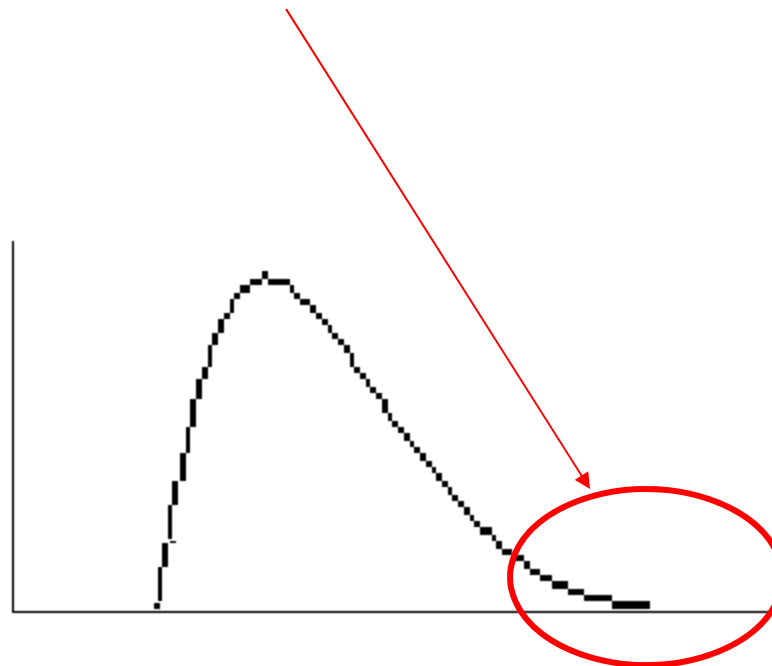
Export Summary Print Summary Edit Layout Best Fit Columns Hide Selected Exit

Sample Name	Material	Source	Lot	D(v,0.1)	D(v,0.5)	D(v,0.9)
Sample 4	PinnoThin TG Powde Herbalife			0.052	0.052	0.052
Sample 4	PinnoThin TG Powde Herbalife			0.052	0.052	0.052
Sample 4	PinnoThin TG Powde Herbalife			0.052	0.052	0.052
Sample 4	PinnoThin TG Powde Herbalife			0.045	0.045	0.045
Sample 4	PinnoThin TG Powde Herbalife			0.045	0.045	0.045
Sample 4	PinnoThin TG Powde Herbalife			0.045	0.045	0.045
Sample 4	PinnoThin TG Powde Herbalife			0.040	0.040	0.040
Sample 4	PinnoThin TG Powde Herbalife			0.039	0.039	0.039
Sample 4	PinnoThin TG Powde Herbalife			0.040	0.040	0.040
Sample 4	PinnoThin TG Powde Herbalife			0.048	0.048	0.048
Sample 4	PinnoThin TG Powde Herbalife			0.048	0.048	0.048
Sample 4	PinnoThin TG Powde Herbalife			0.048	0.048	0.048
Sample 4	PinnoThin TG Powde Herbalife			0.045	0.045	0.045
Average				0.046	0.046	0.046
Std. Dev.				0.005	0.005	0.005
CV (%)				9.805	9.805	9.805
USP 429 (30.0, 20.0, 30.0)				PASSED	PASSED	PASSED

Unique, automatic feature in LA-950 software  
 See Technical Note 169 in Download Center  
 for instructions to use these features

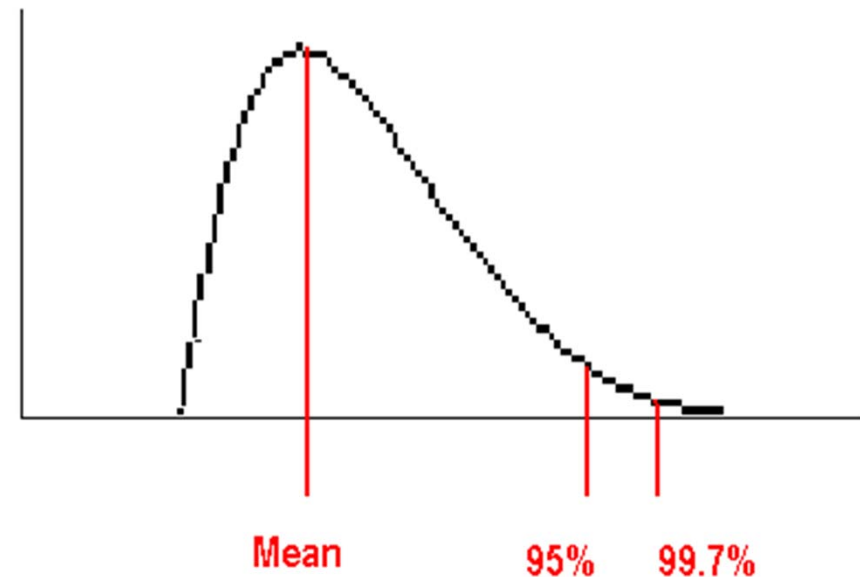
# Distribution extremes

- At a distance of a few standard deviations, non-instrumental errors can dominate



## 2 and 3 standard deviations

- 95% of the distribution is within 2 standard deviations from the Mean
- 99.7% of the distribution is within 3 standard deviations from the mean



# Reproducibility at the extremes

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- If we want the same level of reproducibility at the D99 value as the D50, we need to analyze similar amounts of material in the D99 histogram band

# A better method to monitor extremes

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- Instead of specifying the D95, D99, D99.99, D100, DMax
- Specify the % of material greater than a certain size



# LA-960 Method Expert

The screenshot shows the LA-950 for Windows software interface. A red arrow points to the 'Method Expert' icon in the toolbar. The 'Method Expert' wizard window is open, displaying a welcome message and navigation options.

**LA-950 for Windows - [Result Data View]**

File View Graph Display Conditions Navigator File List Utility Export Option Advanced Function Sampler Help

LA Navigator Overlay memory

☒ Example 1.1  
☒ Example 1.2  
☐ Example 2.1  
☐ Example 2.2  
☐ Example 3.1  
☒ Example 3.2

S.P.Area : 2.6718E+5(cm<sup>2</sup>/cm<sup>3</sup>)  
 Mean Size :  
 Variance :  
 Median Size :  
 Mode Size :  
 Std.Dev. :  
 CV :

Transmittance

Method Expert Wizard

partica LA-950  
**Method Expert**

Welcome to the Method Expert for the HORIBA LA-950 particle size analyzer!

Think of the Method Expert as one big wizard to help you develop a robust method for running any sample with high accuracy and precision. The measurement process is divided into three sections containing simple, guided tests to help you generate great particle size data.

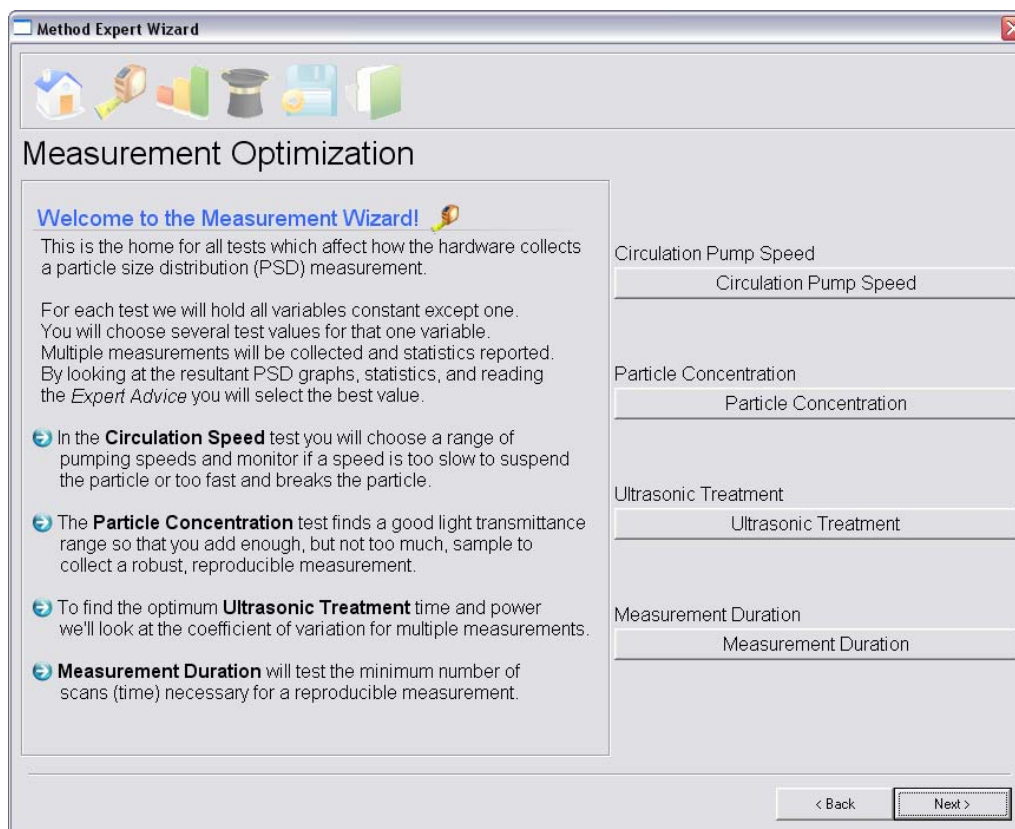
You'll find two special buttons throughout the Method Expert:  
 Click to discover more information about why a particular idea or test is important.  
 Click on the results screen to read HORIBA's *Expert Advice* for choosing the best value for that particular test.

Start Measurement Wizard  
 Start Calculation Wizard  
 Start Automation Wizard

< Back Next >

# Method Expert Hardware

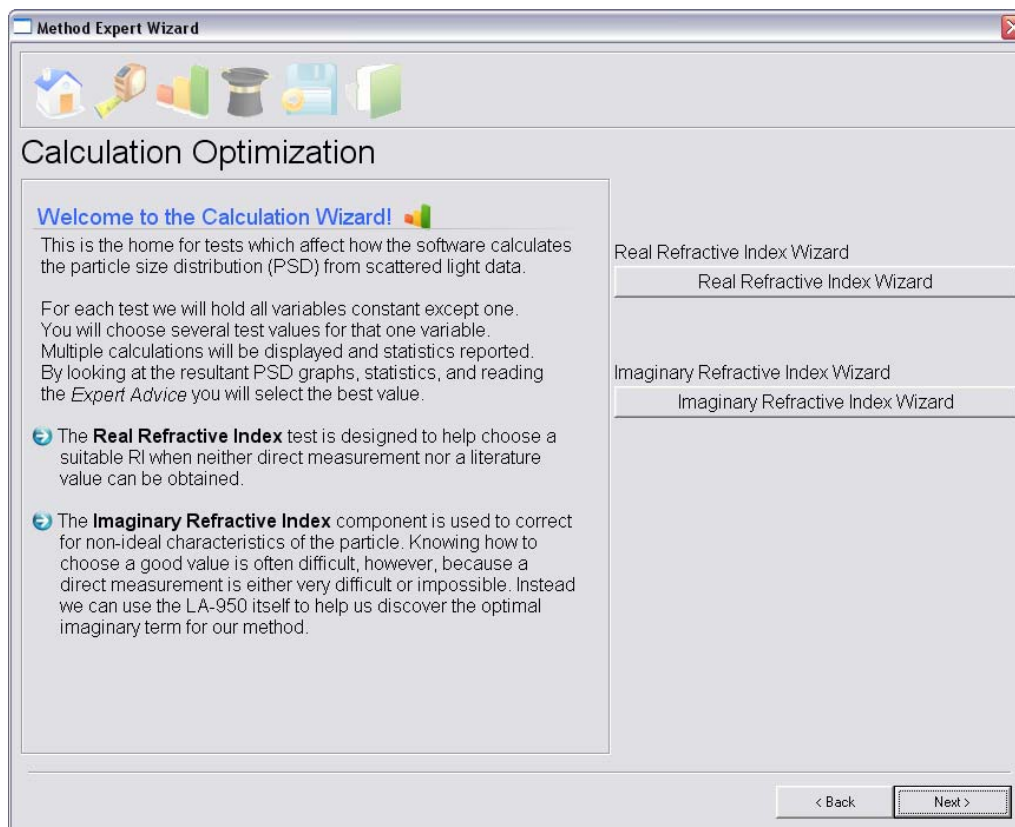
There are four important tests...



Circulation  
Concentration  
Dispersion  
Duration

# Method Expert Calculation

There are two important tests...



Real RI  
Imaginary RI

# LA-960 Method Expert

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**Why is the test important?**

**What does the test do?**

**How will the results be displayed?**

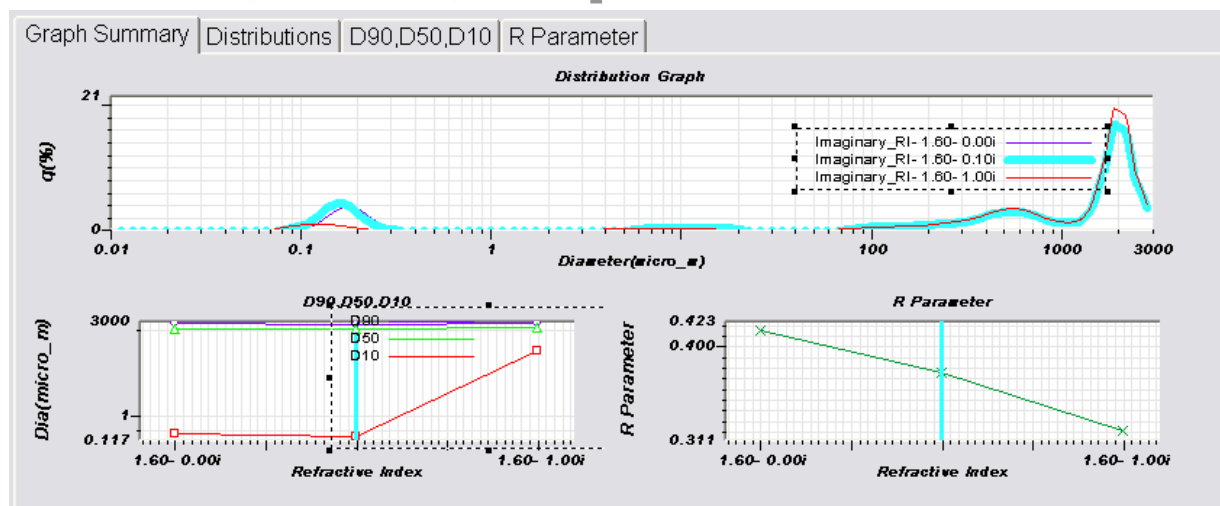
**What is the best value?**

**User selects up to 5 values for testing**

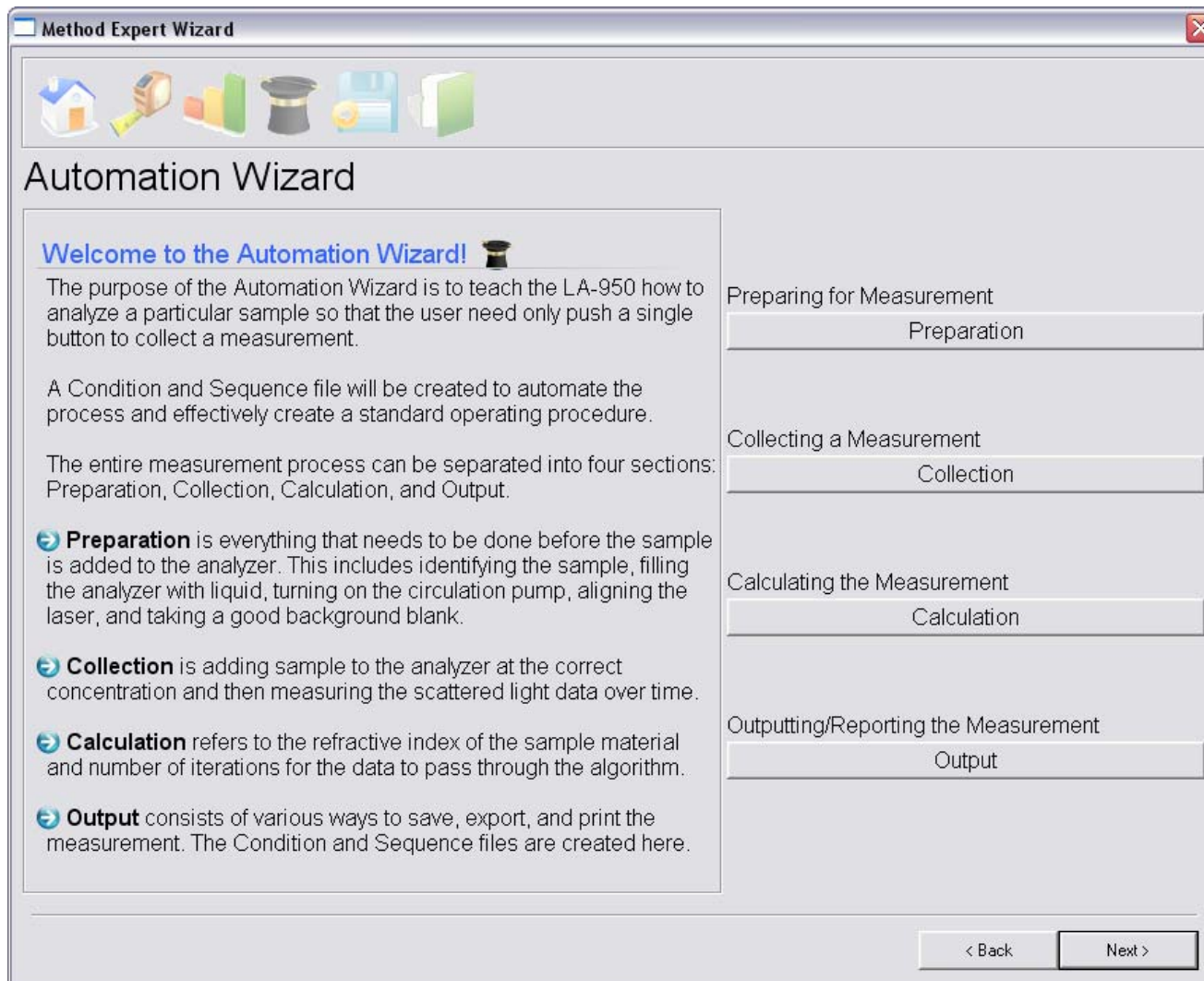
# LA-960 Method Expert

Method Expert guides user to prepare the LA-950 for each test

Results displayed in multiple formats:  
PSD, D50, R-parameter



# LA-960 Method Expert





# LA-960 Method Expert

Method Expert Wizard

Preparation
Collection
Calculation
Output

## Automation Wizard

### Outputting/Reporting the Measurement ?

#### Section Purpose

Remember to click the ? button for more information

The measurement has been collected and calculated and can now be saved, exported, and printed for reporting. The LA-950 was designed to meet a variety of customer preferences, so there are many ways to perform these tasks.

Once the reporting setup is finished, simply name the Condition and Sequence files used to run this method.

Step 4. Give this Expert Method a unique, descriptive name.

(This name is used as the output sequence file name)

?

☒ Use same name for saving the condition file.

Step 5. Input condition file name.

?

Step 6. Push save button.

This wizard is temporarily closed,  
and the sequence file and condition file are saved.

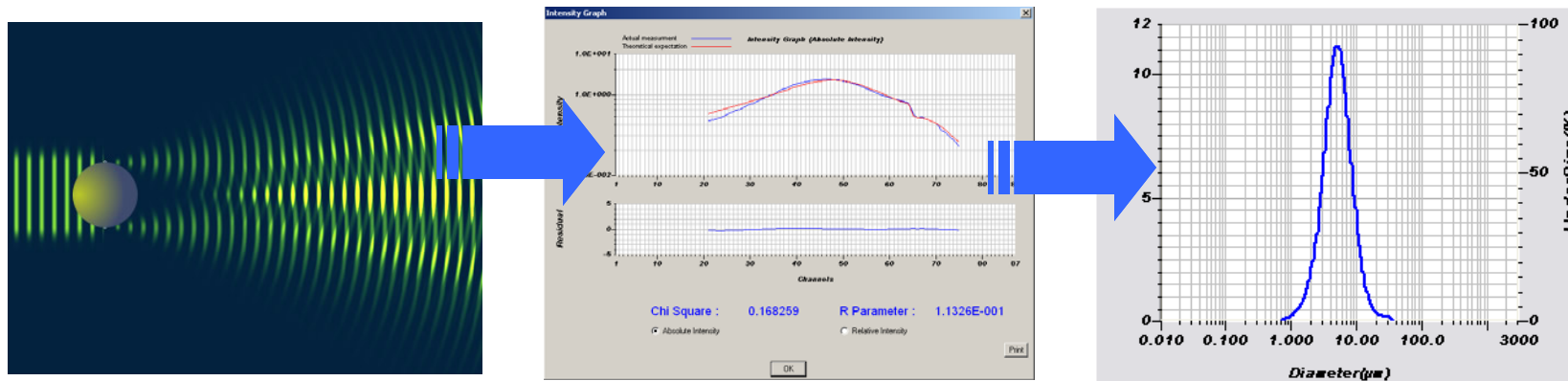
Save Sequence and Condition

< Back
Next >



# Intensity Graph

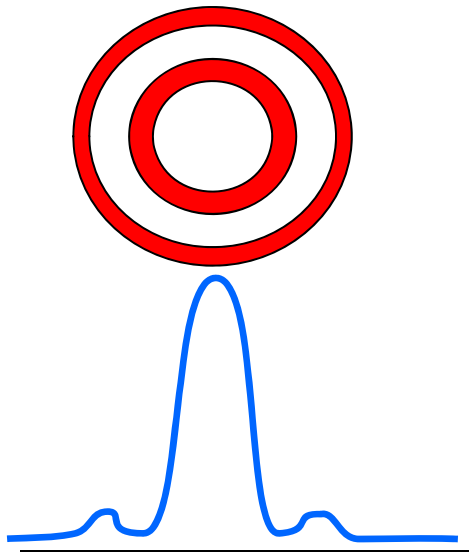
- Diffraction analyzer measures light scattering pattern, algorithm transforms this into a particle size distribution



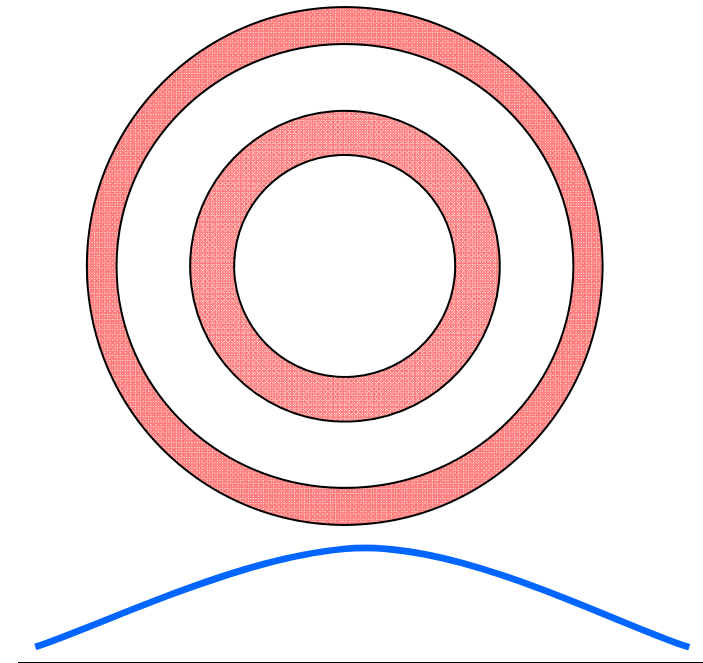
# Size affects intensity

## ■ LARGE PARTICLE:

- Low angle scatter
- Large signal



**Narrow Pattern - High intensity**



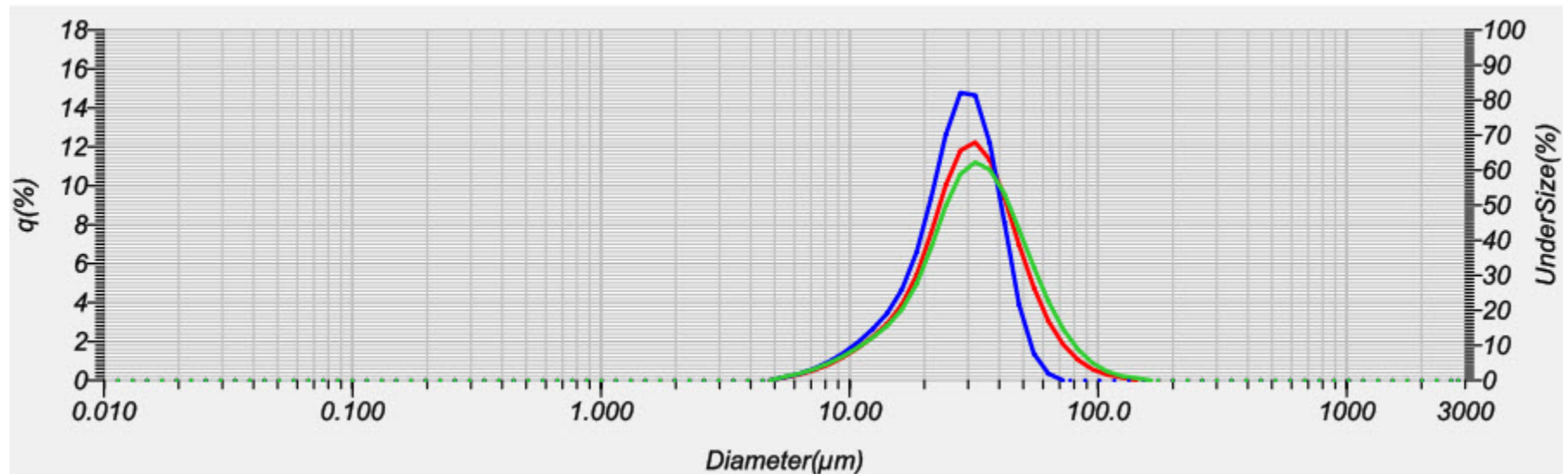
**Wide Pattern - Low intensity**

## ■ SMALL PARTICLE:

- High Angle Scatter
- Small Signal

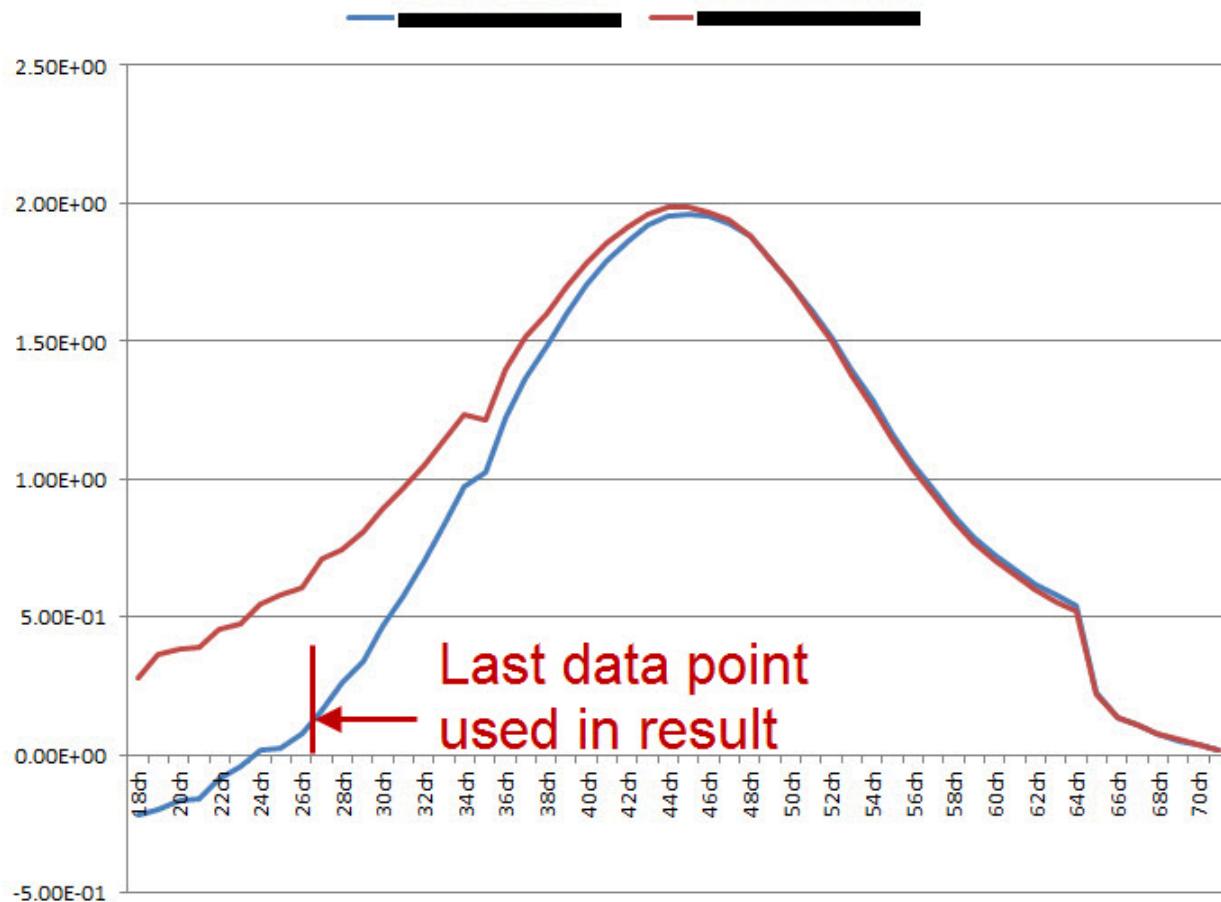
# Intensity Graph

- One way to use the Intensity Graph
  - Two results, one good and one bad



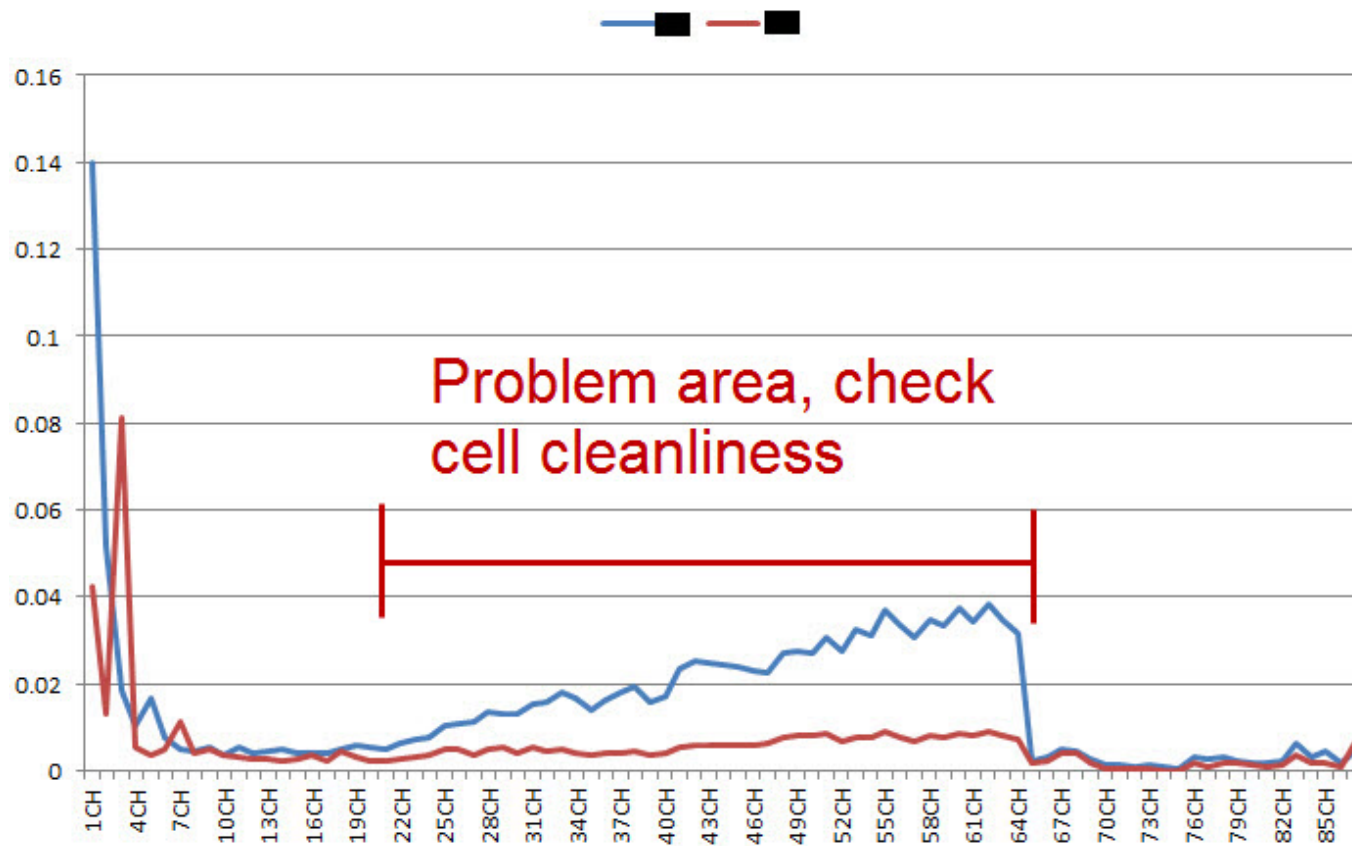
# Intensity Graph

■ Pull up the tool and compare



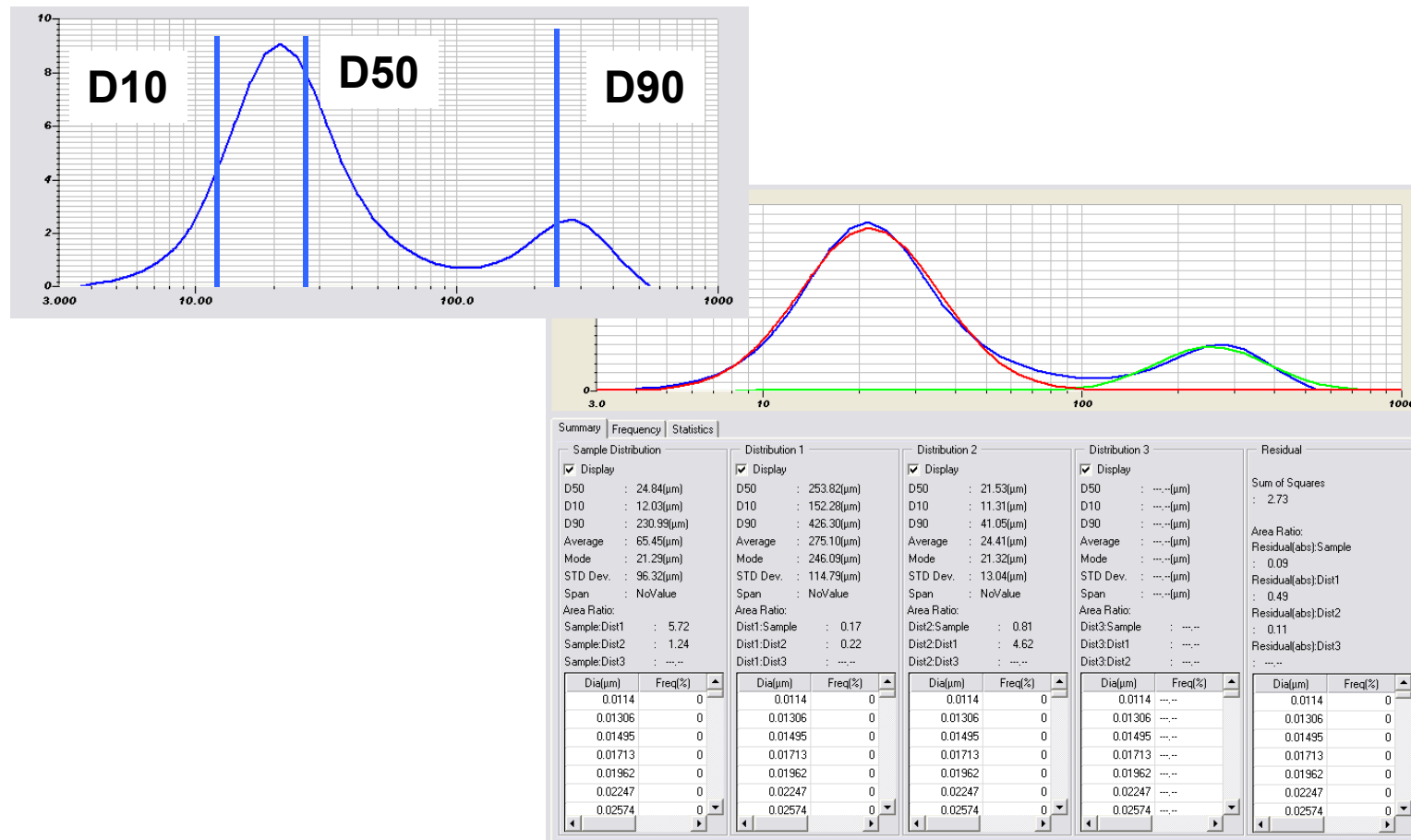
# Intensity Graph

- Need to explain difference in scattering
- Try other tools, i.e. Blank Check



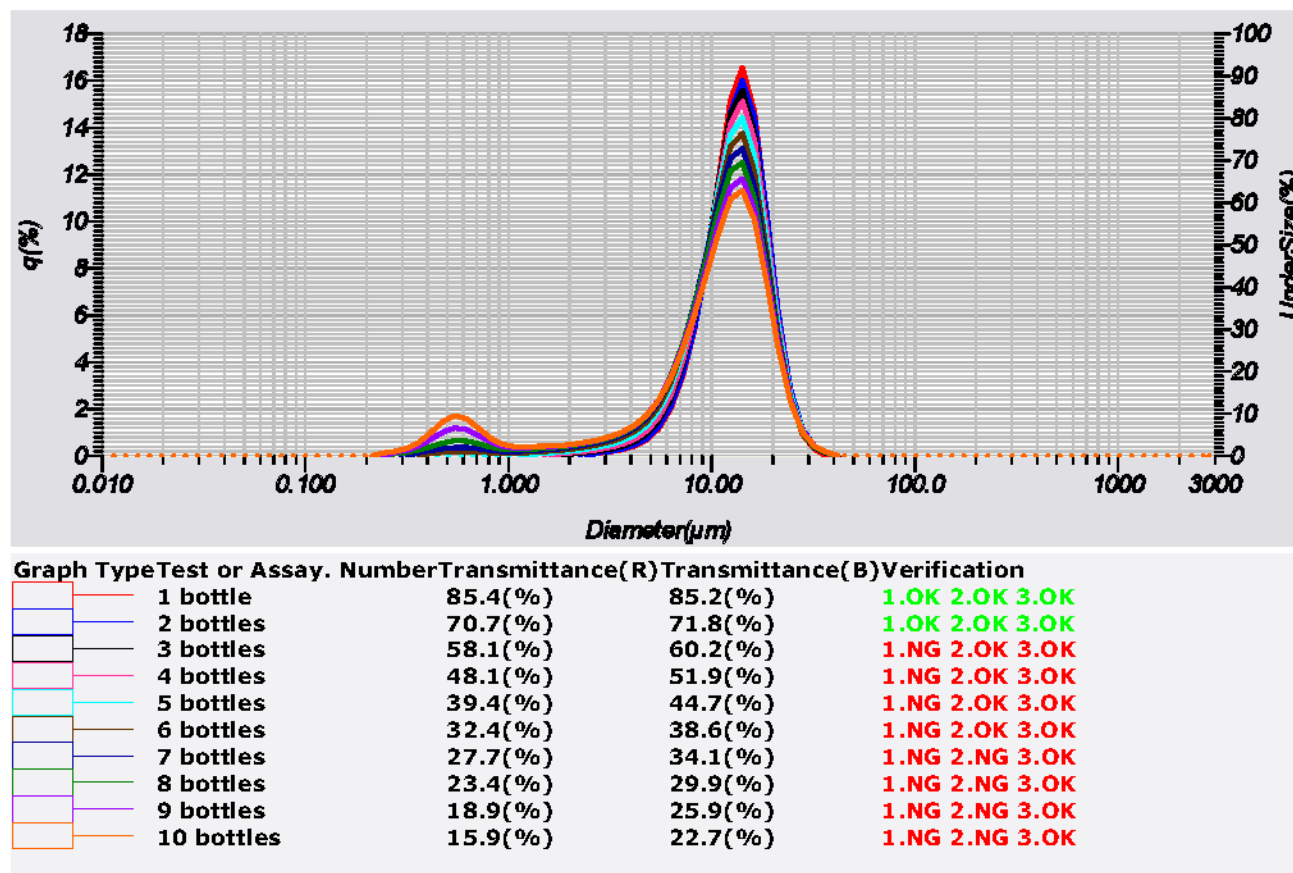
# Multi-modal distributions

Multiple peaks can be better described individually



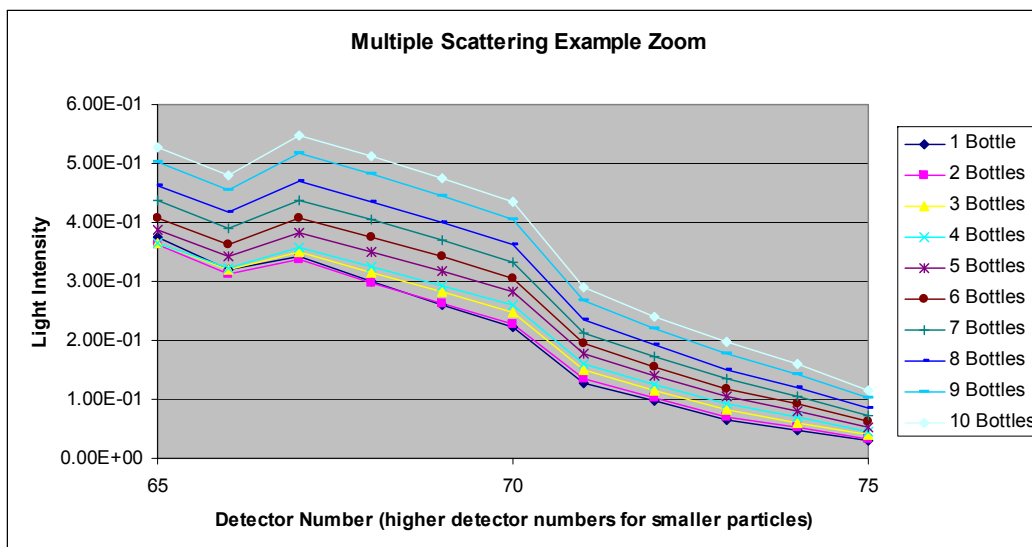
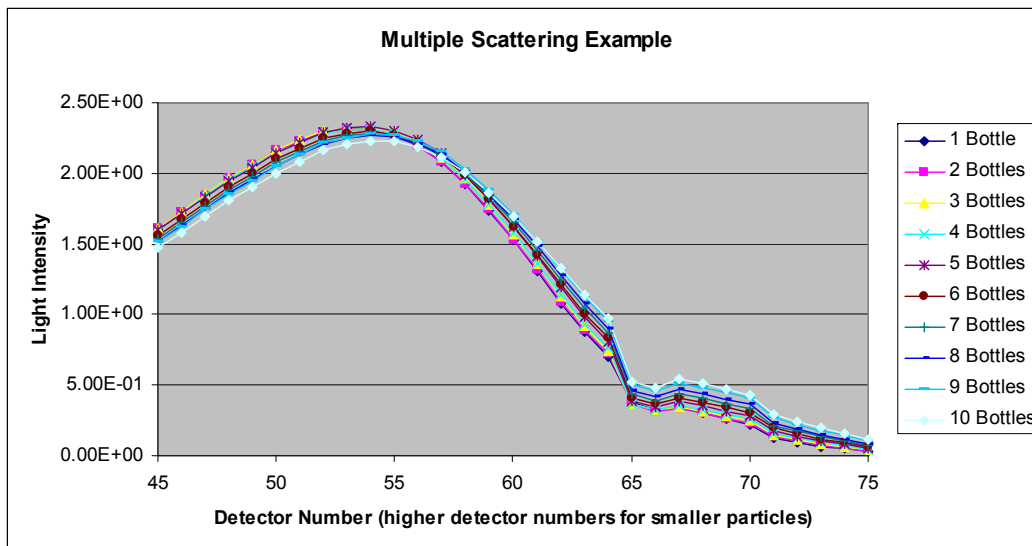
# Multiple scattering

Watch for finer “particles” appearing with increasing concentration





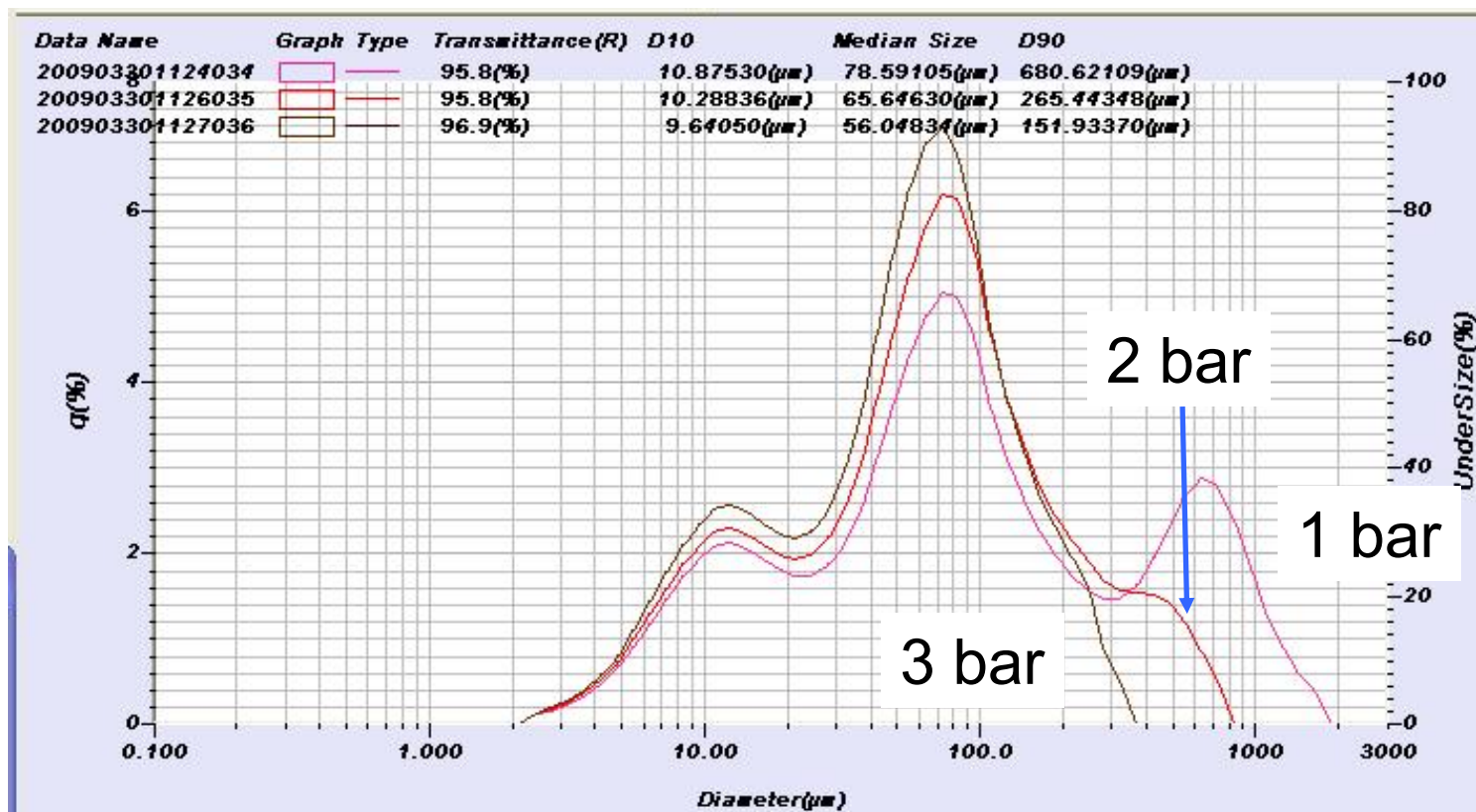
# Multiple scattering





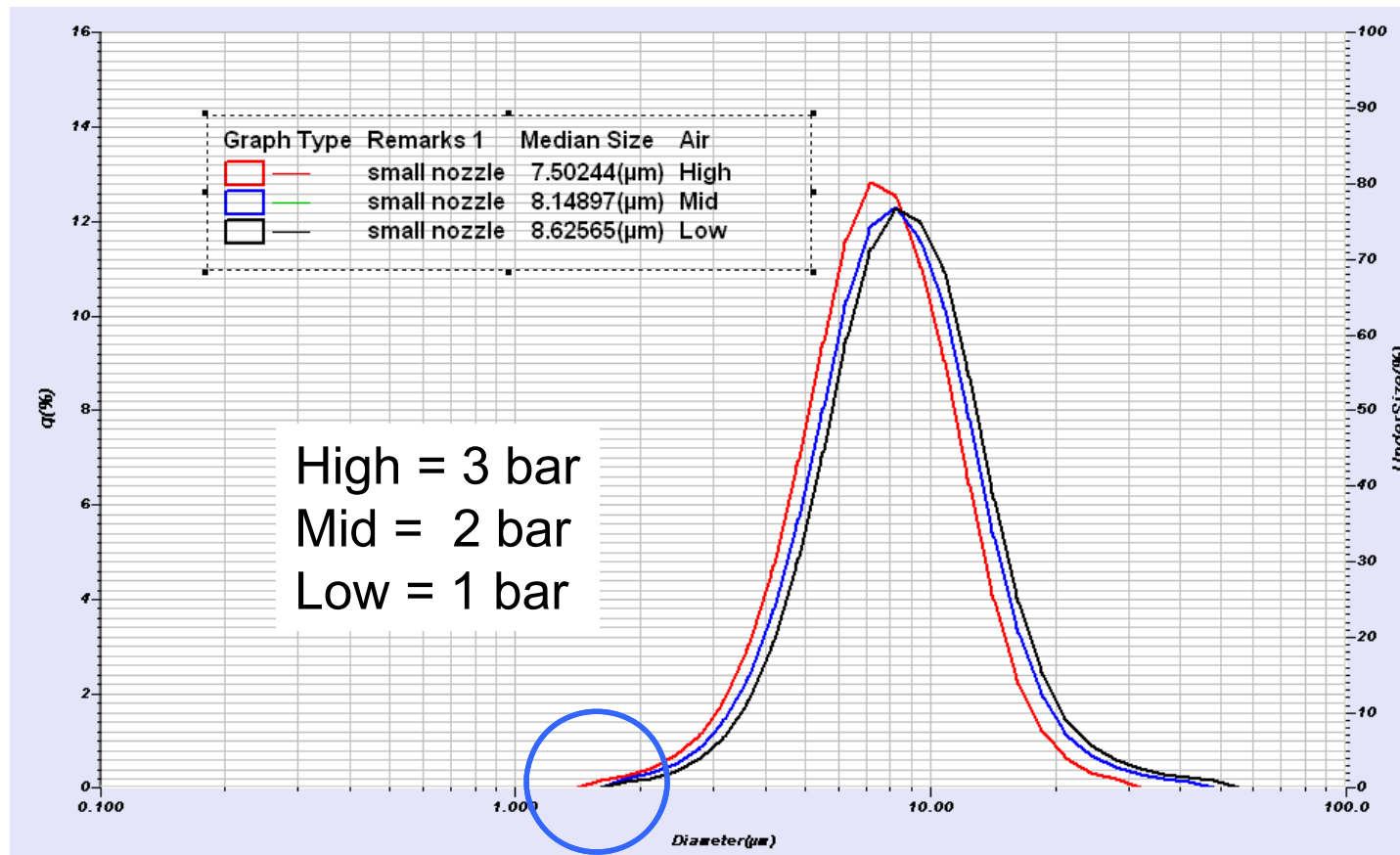
# Dispersing agglomerates

Watch for no change in coarsest particles with changing energy



# Breaking particles

Watch for finer particles being created with increasing energy



**Thank you**

ありがとうございました

Dziękuję

ขอบคุณครับ

谢谢

Cảm ơn

Gracias

اشْكُرْ

Σας ευχαριστούμε

धन्यवाद

Grazie

Tacka

Danke

Merci

நன்றி

감사합니다

Большое спасибо

Obrigado

おかしく

*Omoshiro Okashiku*

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**ian.treviranus@horiba.com**

← Talk to us, ask questions  
**labinfo@horiba.com**

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**HORIBA Scientific**

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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

### Request Information

### Quick Request

E-mail us

### Particle Size Essentials

eBook

### Newsletter

Sign Up

### Download Center

More Info

### Member login

Please enter your e-mail address and password in order to login on to www.horiba.com. We may ask you later to answer additional questions if you are the first time to this area.

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Address:   
Password: