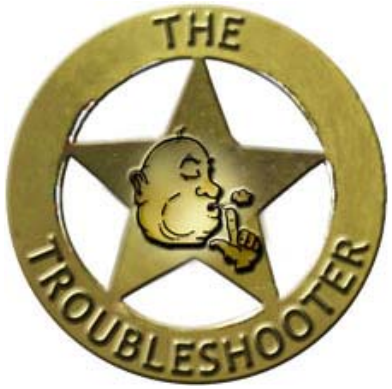
Troubleshooting Laser Diffraction Particle Size Results



Ian Treviranus
ian.treviranus@horiba.com
www.horiba.com/us/particle

What we'll talk about

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

Additional resources

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

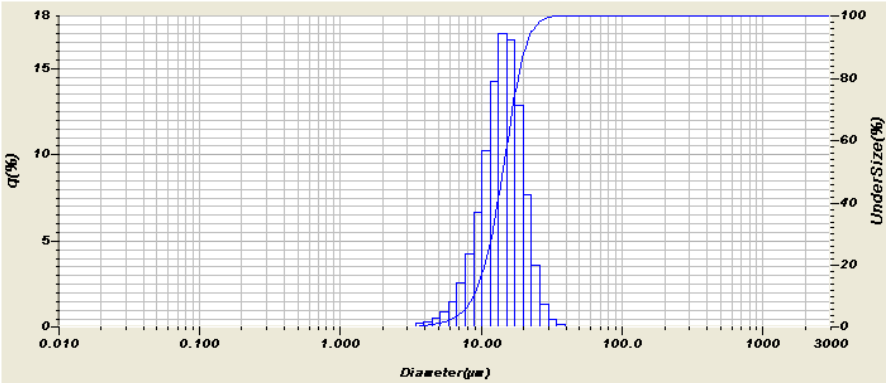
Typical workflow

- 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

Run standard material

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

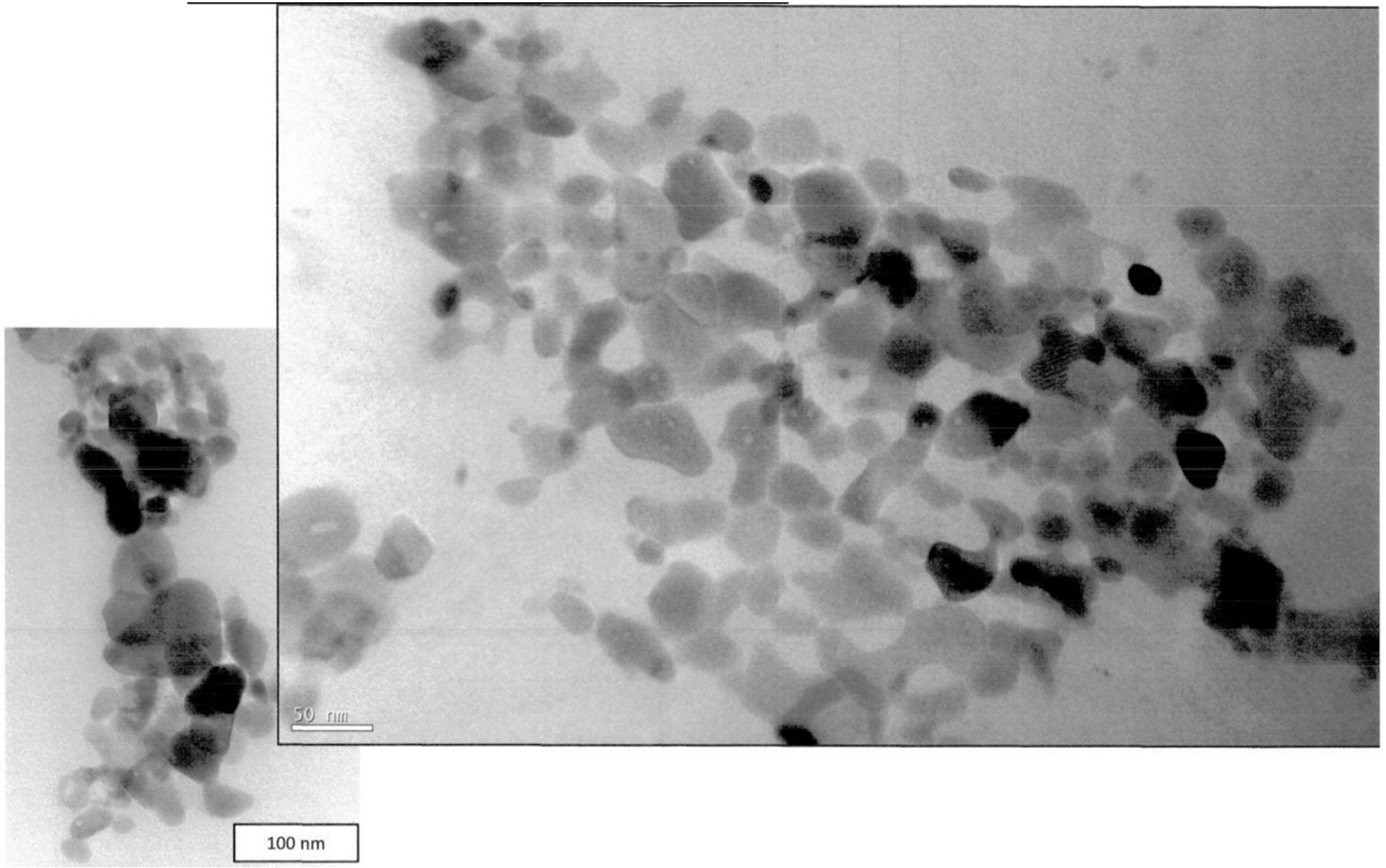
PS202 (3-30µm)	D10	D50	D90
Standard Value (µm)	9.14	13.43	20.34
Uncertainty (µm)	0.86	0.86	1.44
ISO standard error	5%	3%	5%
Lower limit (µm)	7.866	12.193	17.955
Measured Result (µm)	9.721	13.916	18.959
Upper Limit (µm)	10.500	14.719	22.869



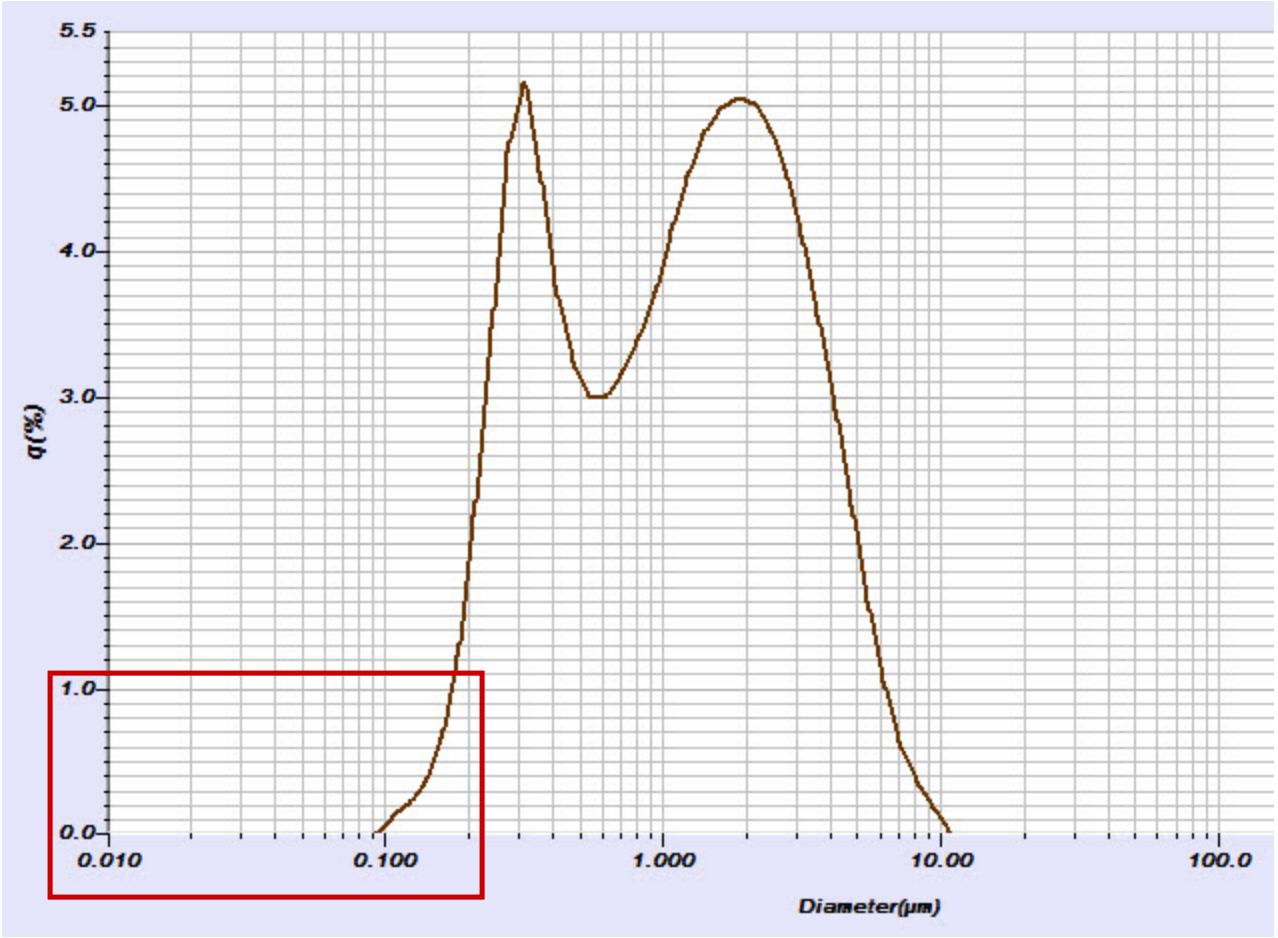
Compatible technologies

- 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

What we'll talk about

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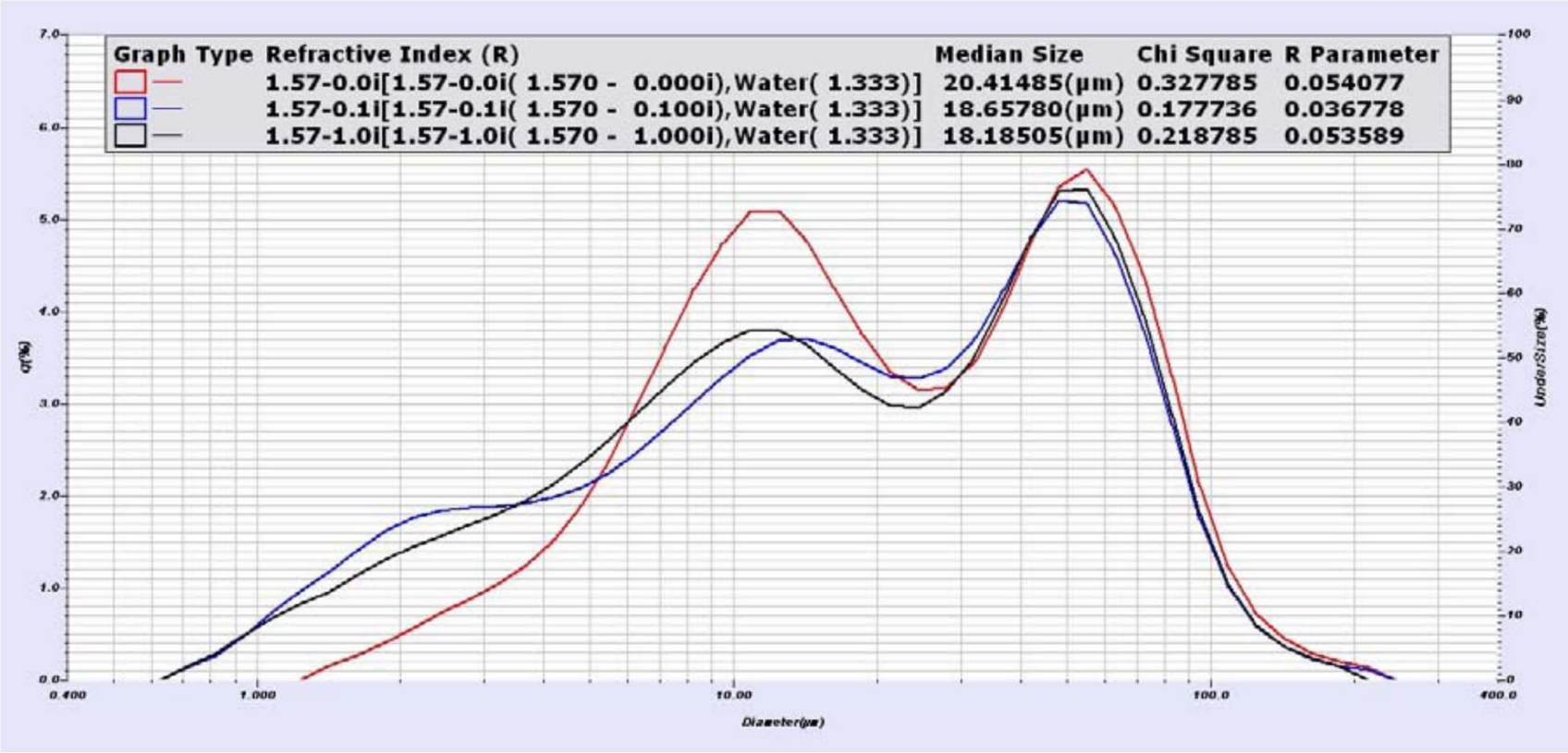
Compare conditions

- 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

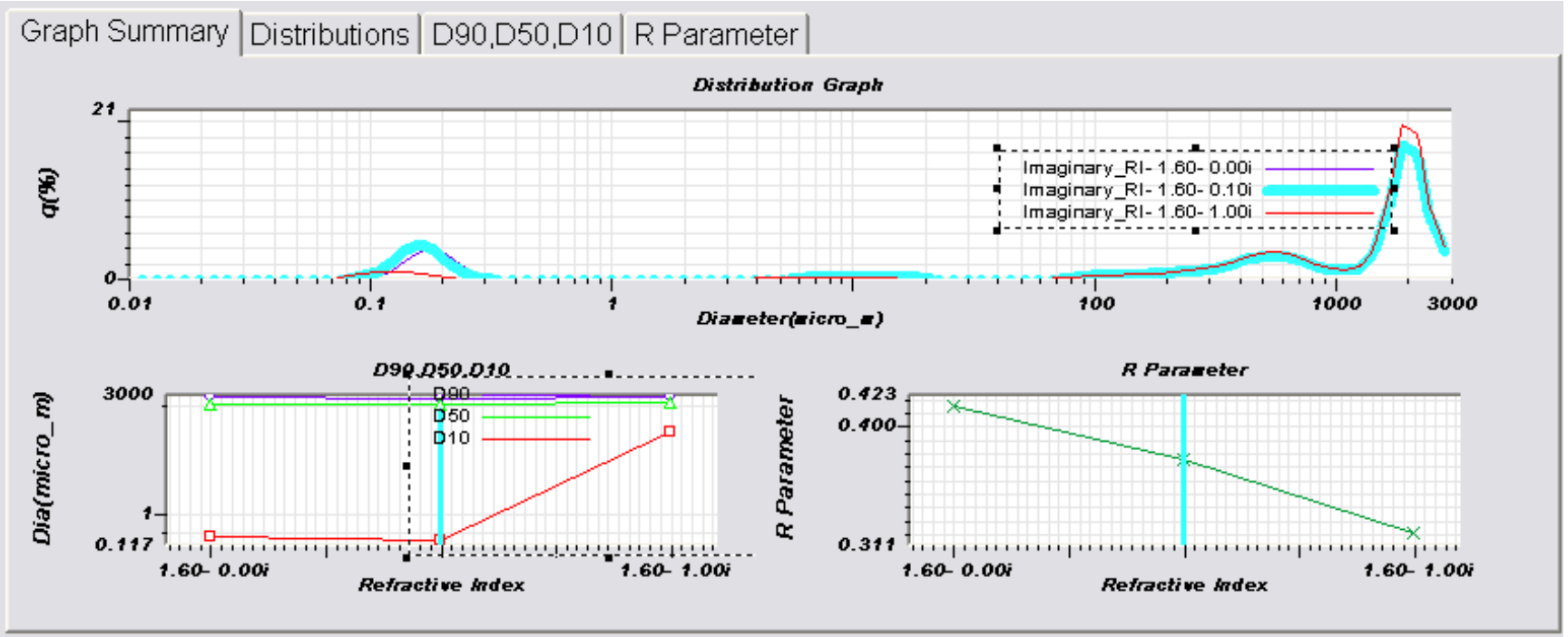
Refractive index

- Seemingly minor differences calculate different distributions



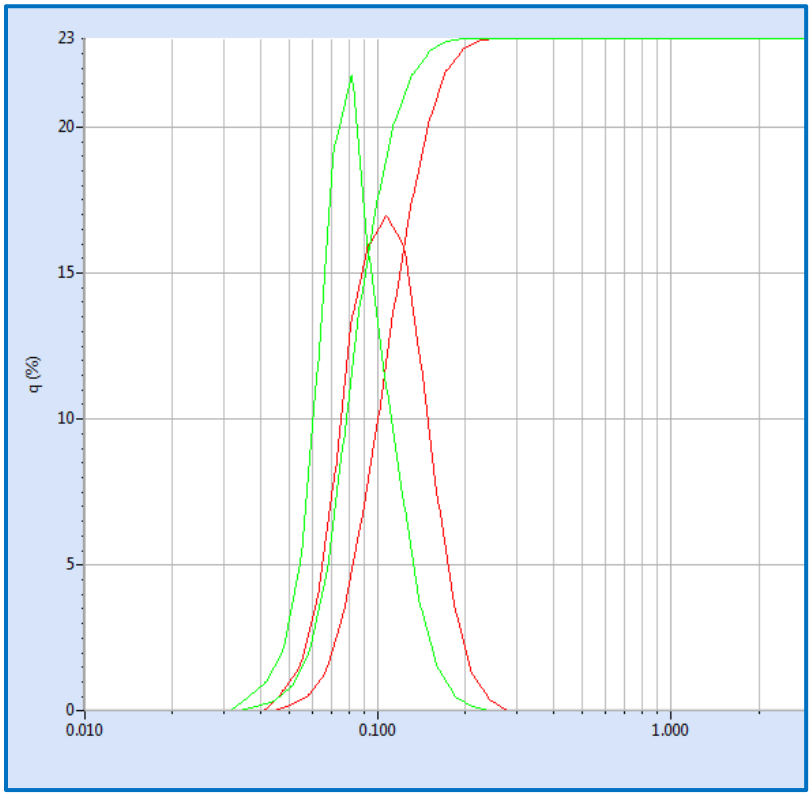
Refractive index

- Compare easily with LA-960 Method Expert software



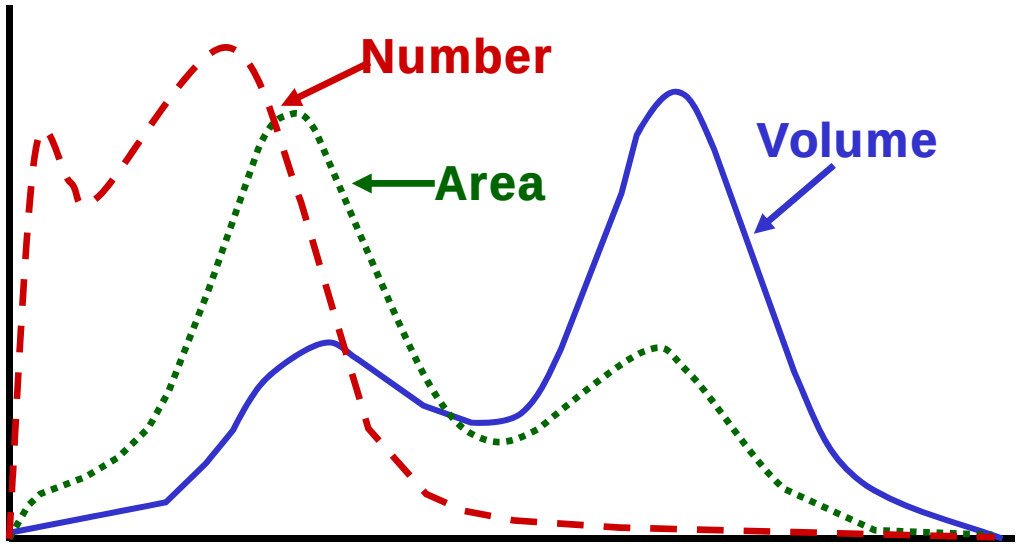
Refractive index

- 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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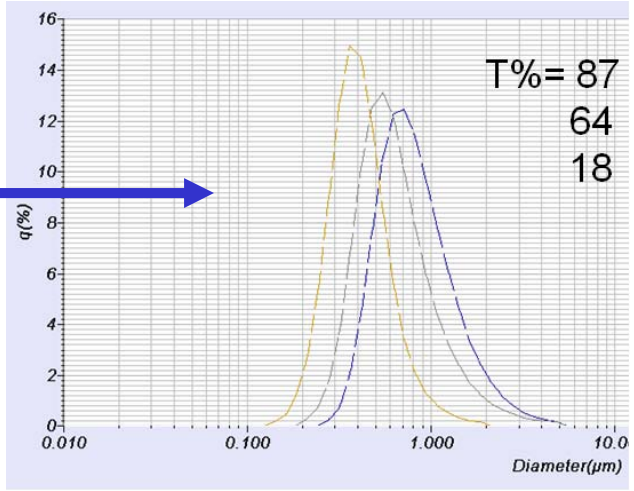
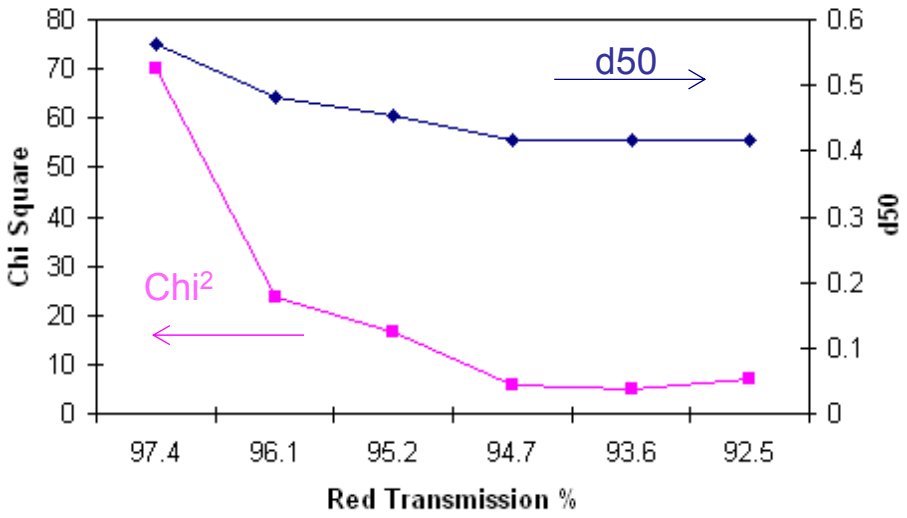
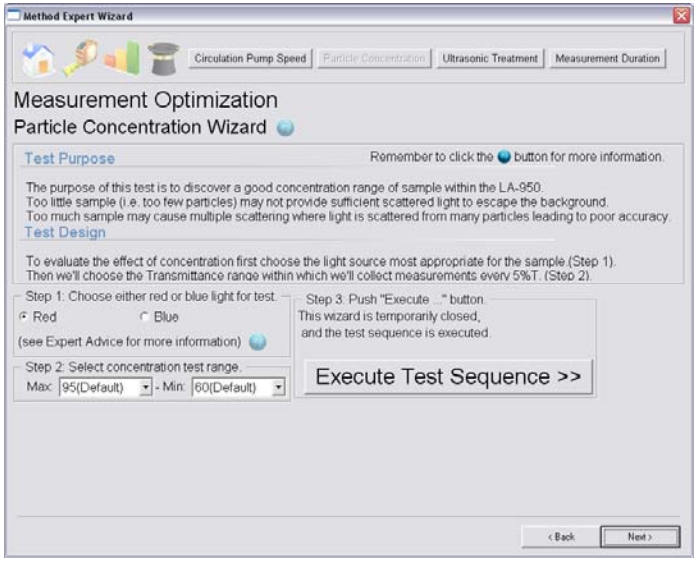
Concentration

- 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



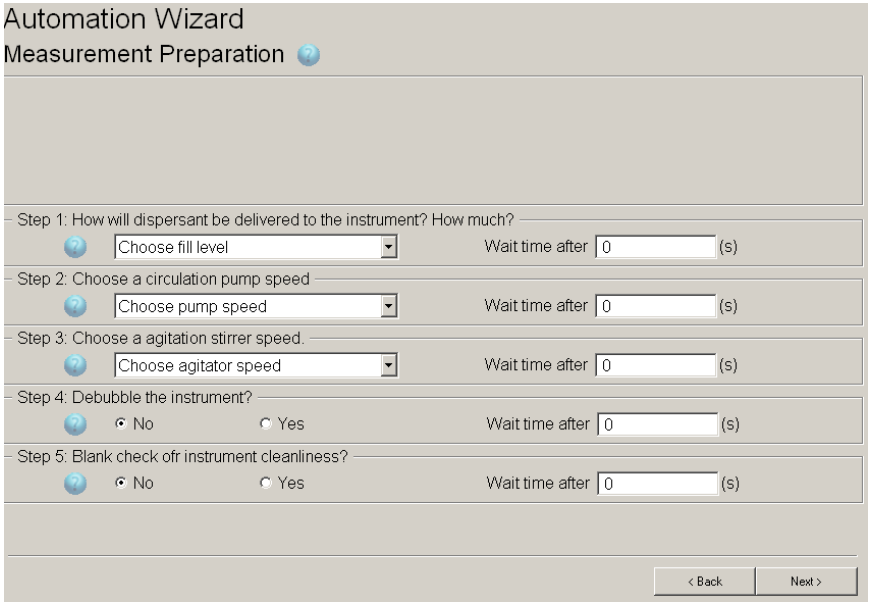




Pump & stirrer

- 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	$D_{mean\ (nm)}$	D_{10} (nm)	D_{90} (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: 2		Circulation: 2	

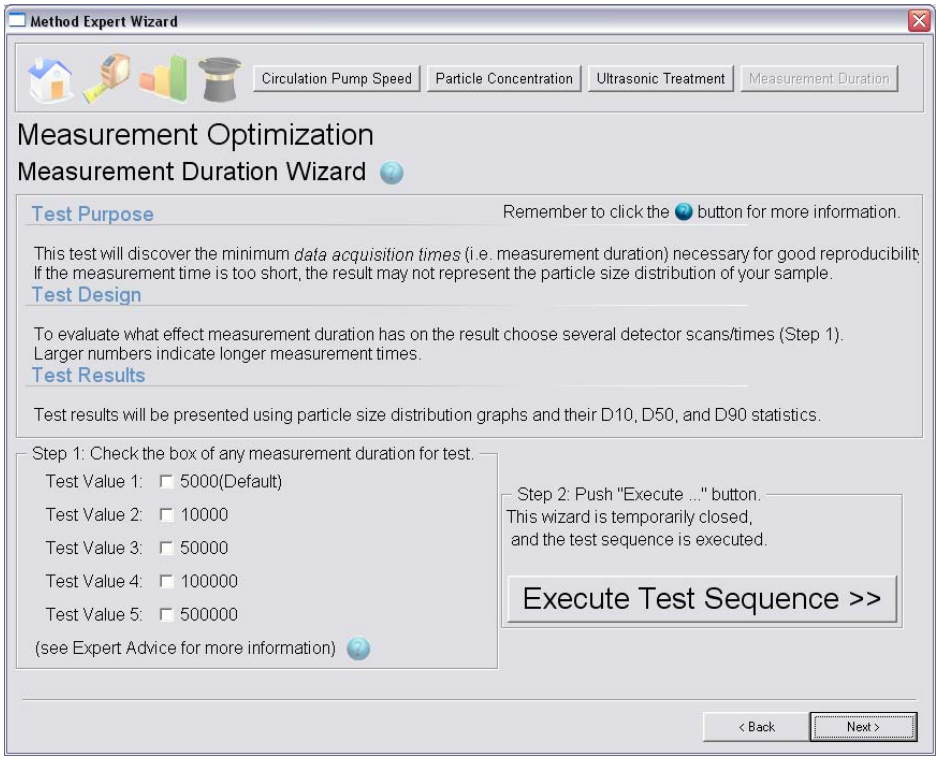


Ultrasonic dispersion

- 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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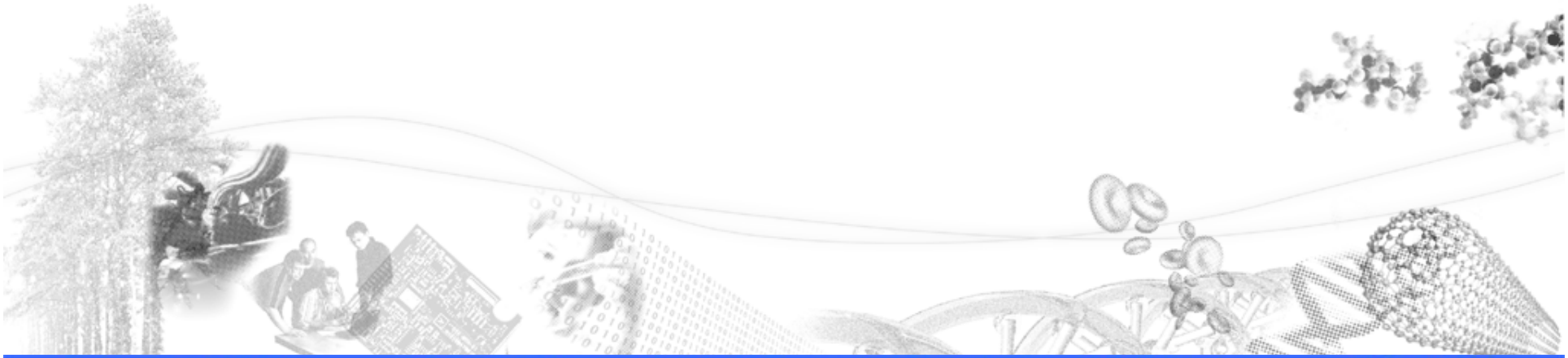
Choosing Parameters



Choosing good statistics

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

The basis for reliable data

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

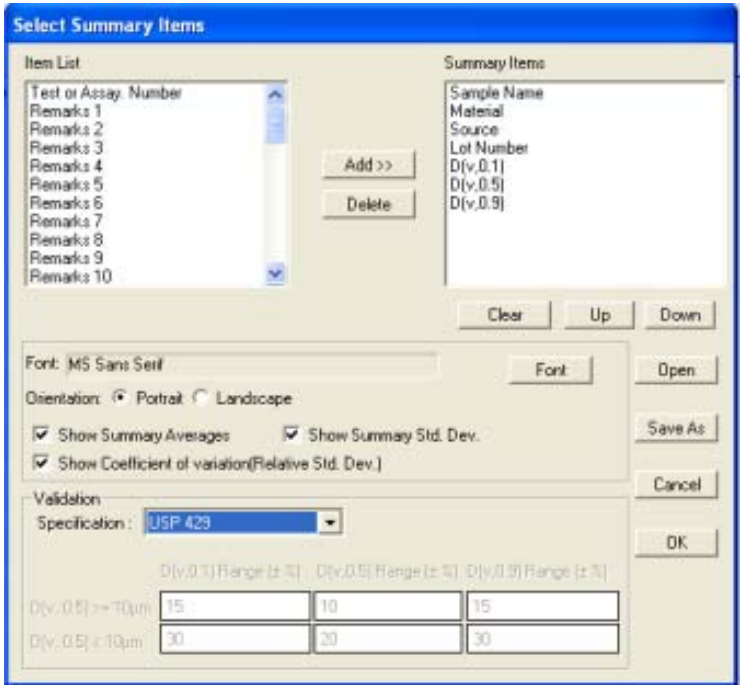
USP <429>
- COV < 10% at Median (D50)
- COV < 15% at D10 and D90

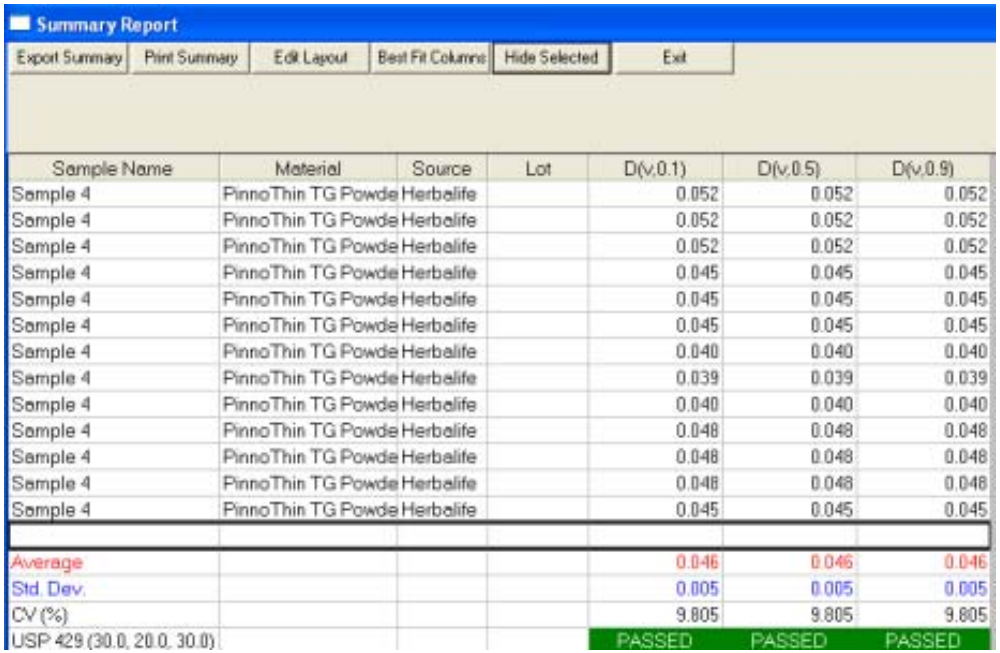
COV = 100 * (StDev / Mean)

Note: All limits double when D50 < 10 µm

Note: Must acquire at least 3 measurements from unique samplings

Calculation automation



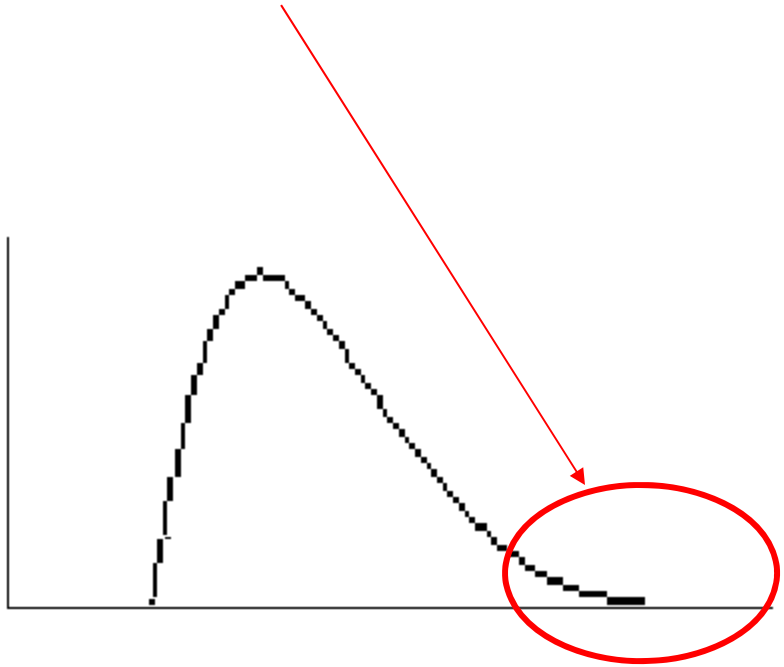


Sample Name	Material	Source	Lot	D(v,0.1)	D(v,0.5)	D(v,0.9)
Sample 4	PinnoThin TG Powde	Herbalife		0.052	0.052	0.052
Sample 4	PinnoThin TG Powde	Herbalife		0.052	0.052	0.052
Sample 4	PinnoThin TG Powde	Herbalife		0.052	0.052	0.052
Sample 4	PinnoThin TG Powde	Herbalife		0.045	0.045	0.045
Sample 4	PinnoThin 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

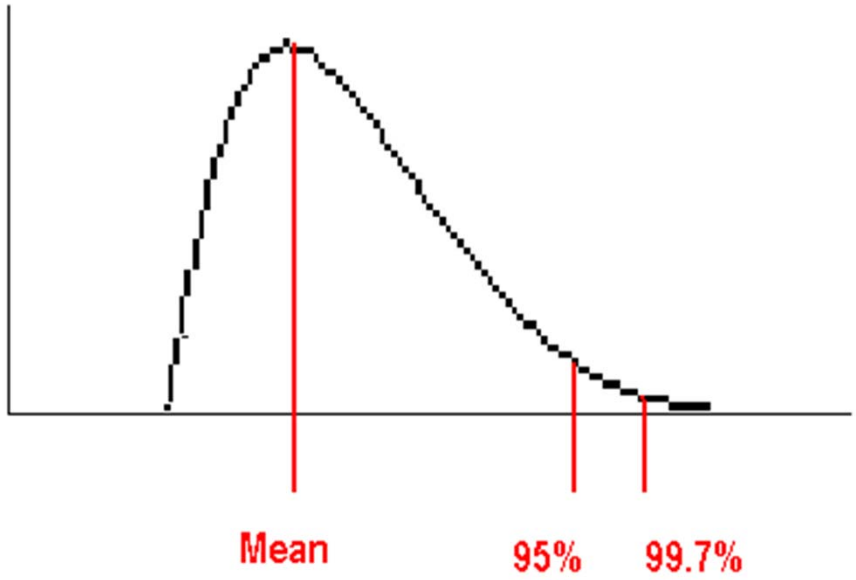


2 and 3 standard deviations

- 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



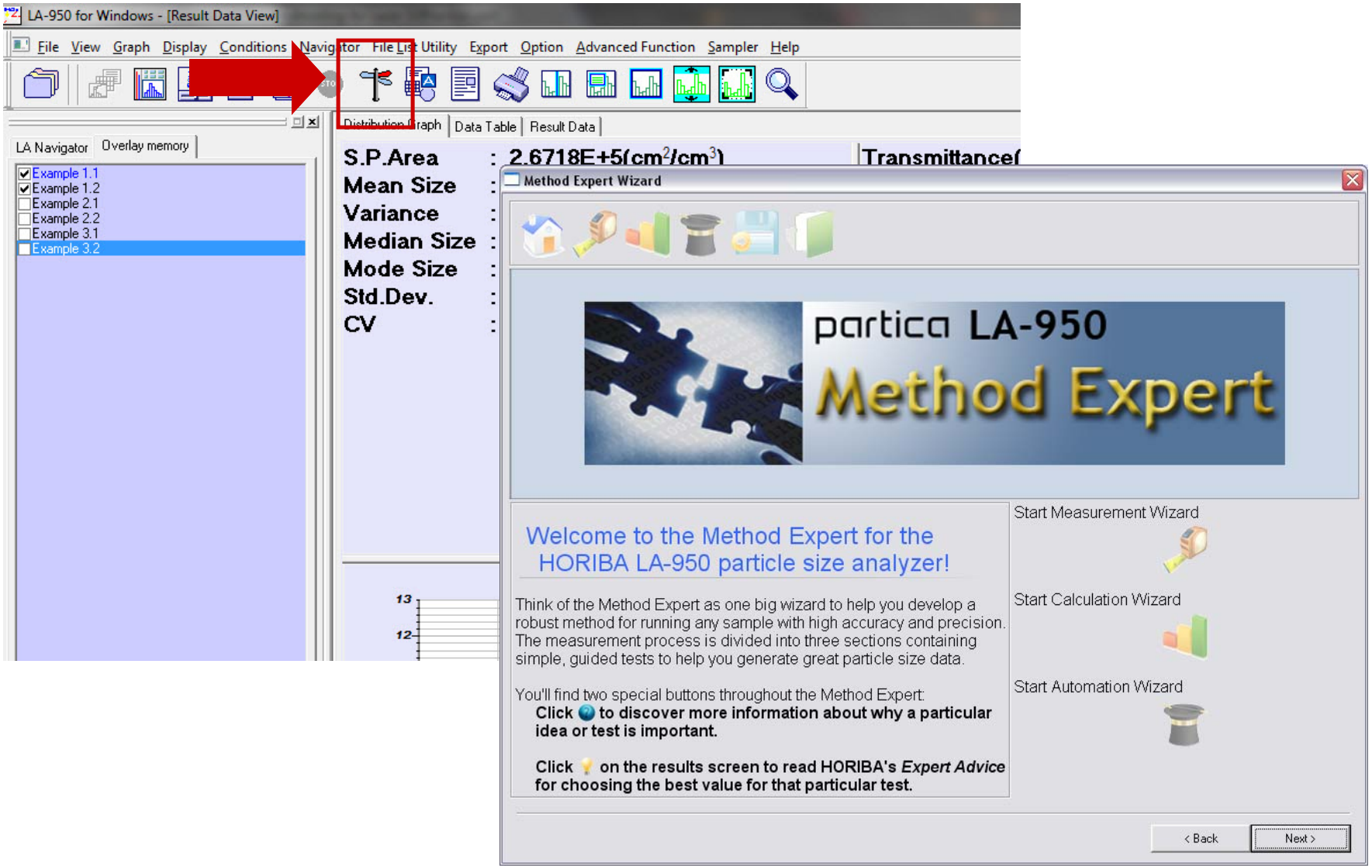
Reproducibility at the extremes

- 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

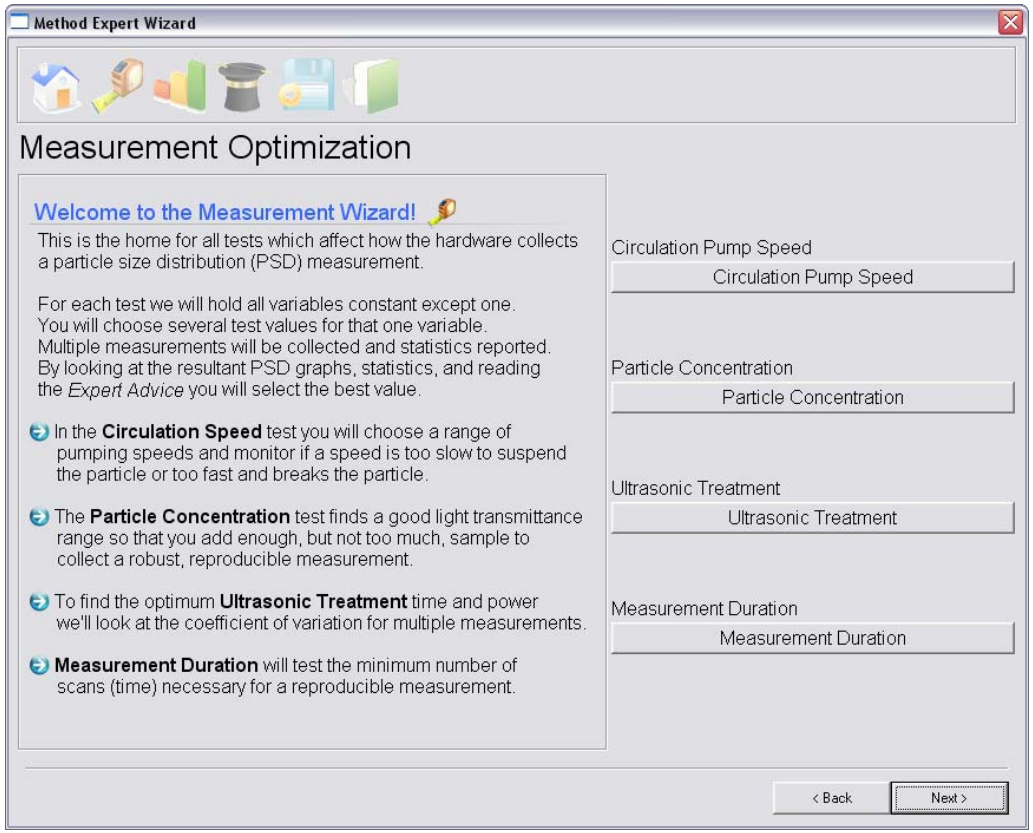
- Instead of specifying the D95, D99, D99.99, D100, DMax
- Specify the % of material greater than a certain size

LA-960 Method Expert



Method Expert Hardware

There are four important tests…



Circulation

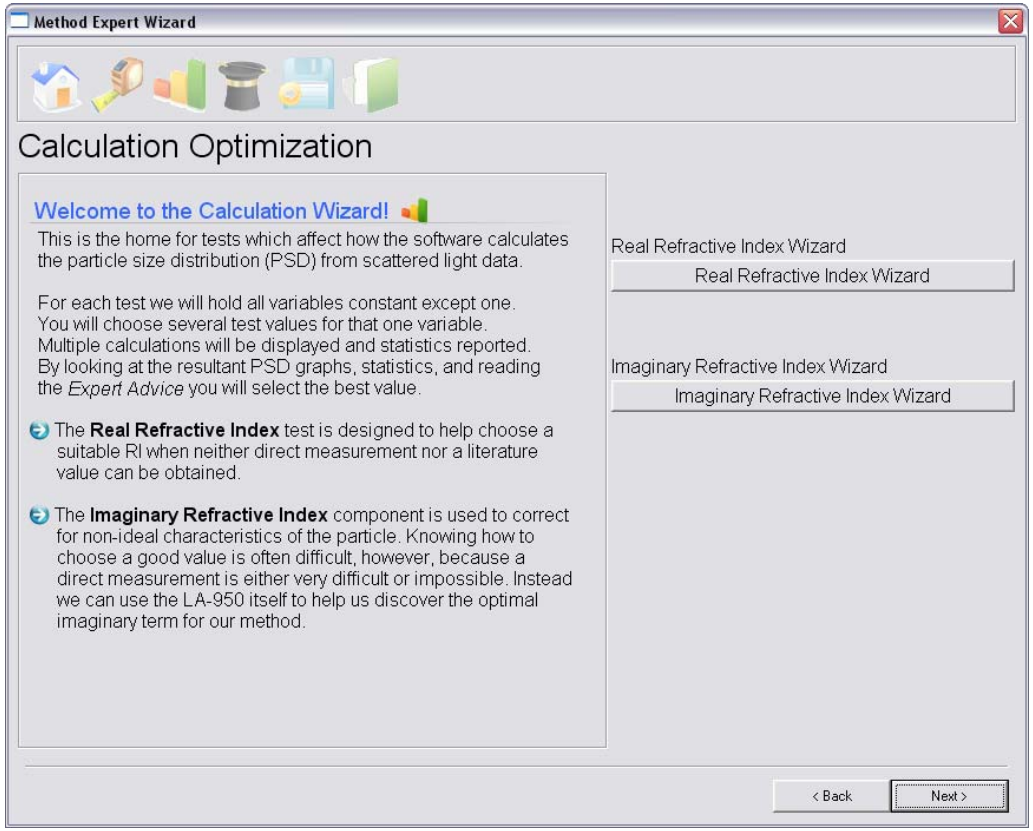
Concentration

Dispersion

Duration

Method Expert Calculation

There are two important tests…



Real RI

Imaginary RI

LA-960 Method Expert

Why is the test important?

What does the test do?

How will the results be displayed?

What is the best value?

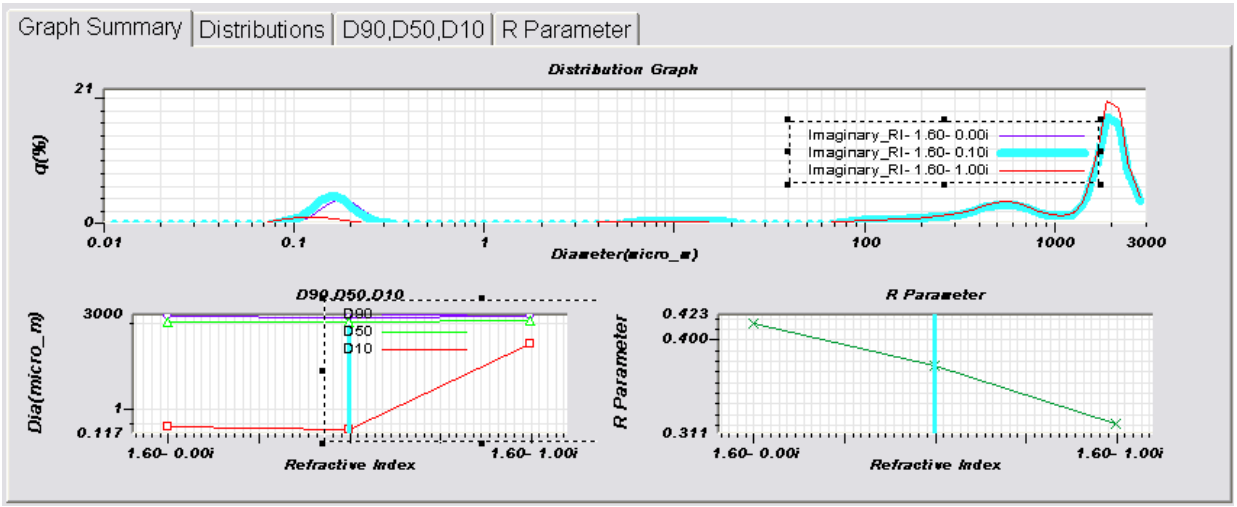
User selects up to 5 values for testing

LA-960 Method Expert

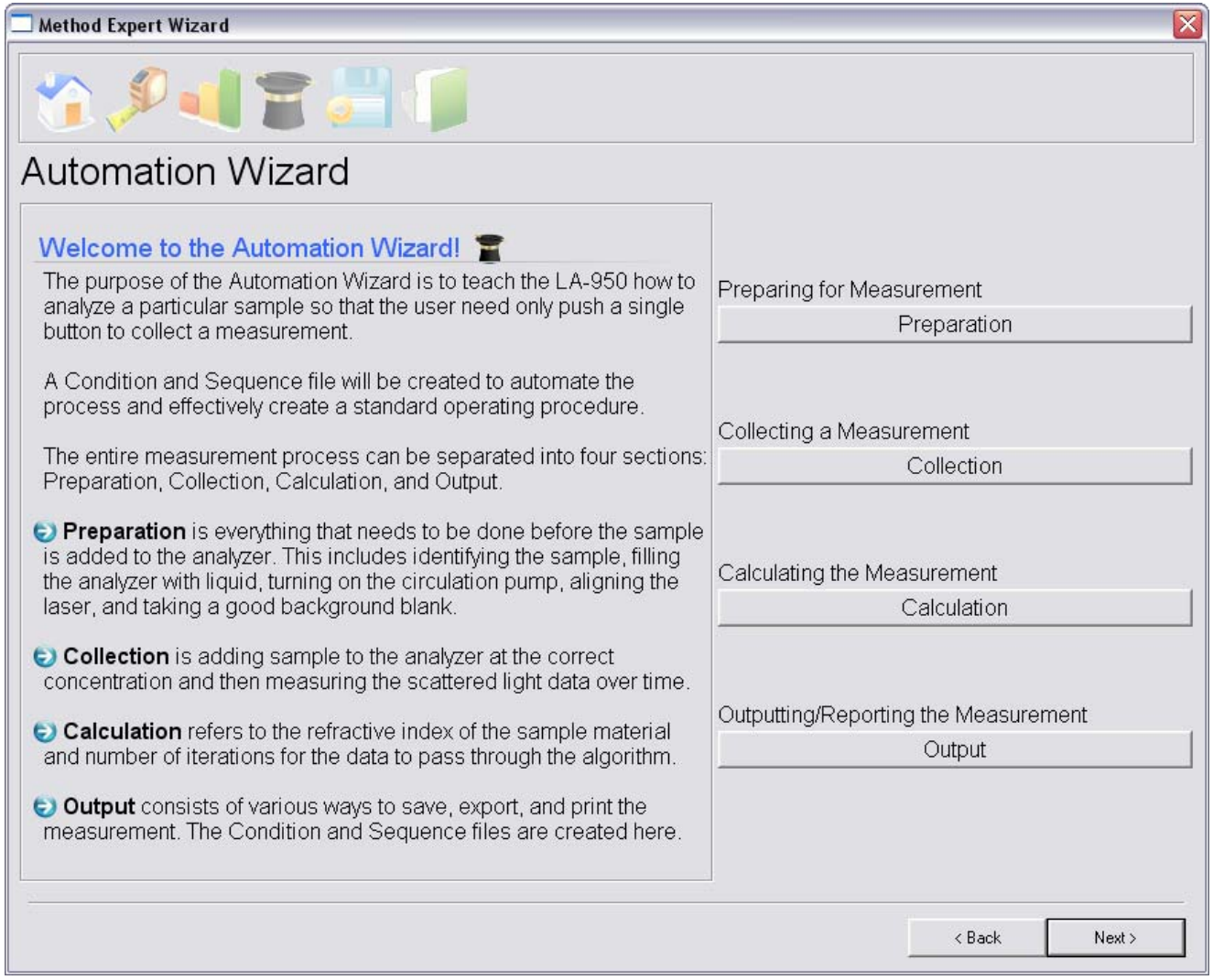
Method Expert guides user to prepare the LA-950 for each test

Results displayed in multiple formats:

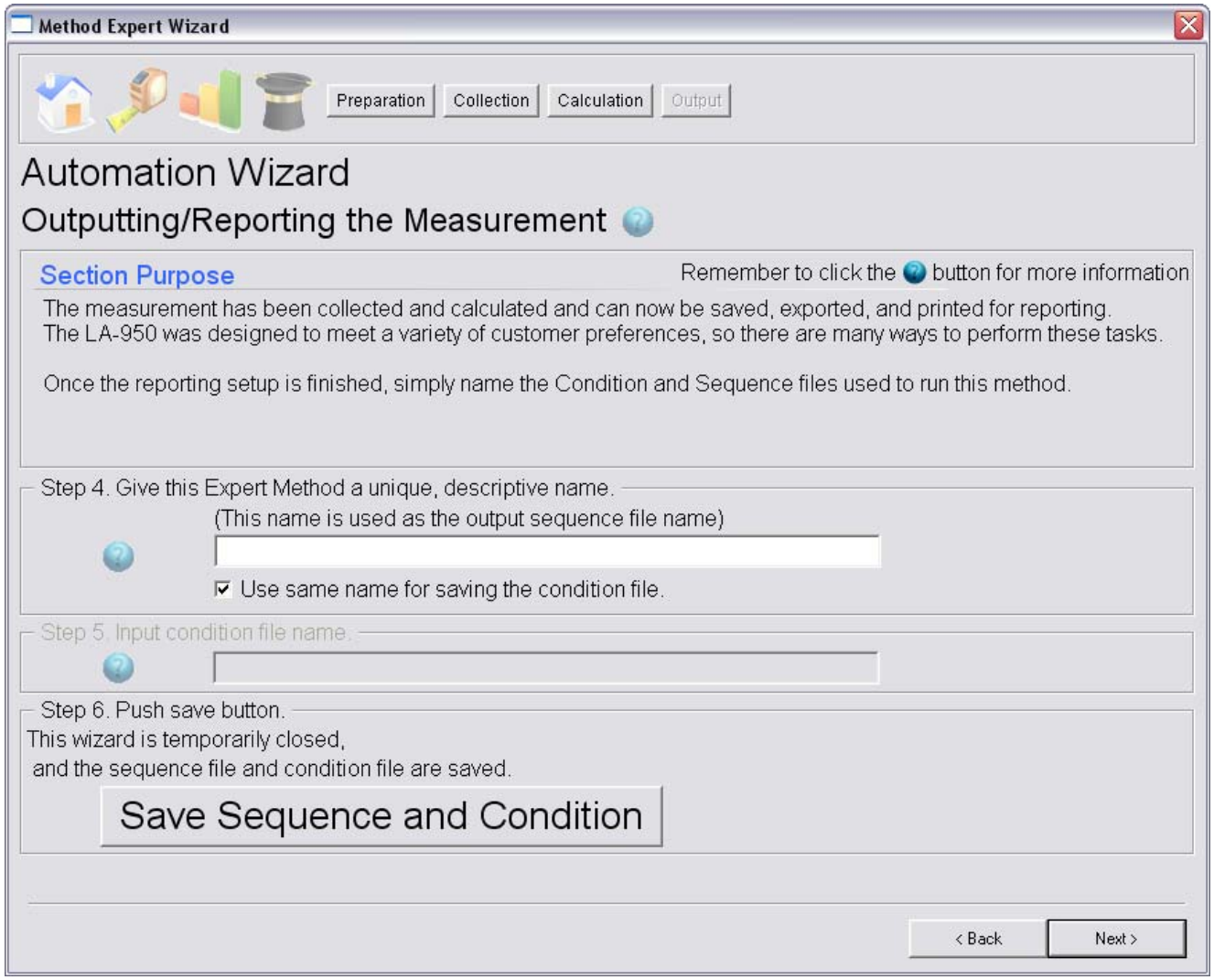
PSD, D50, R-parameter



LA-960 Method Expert

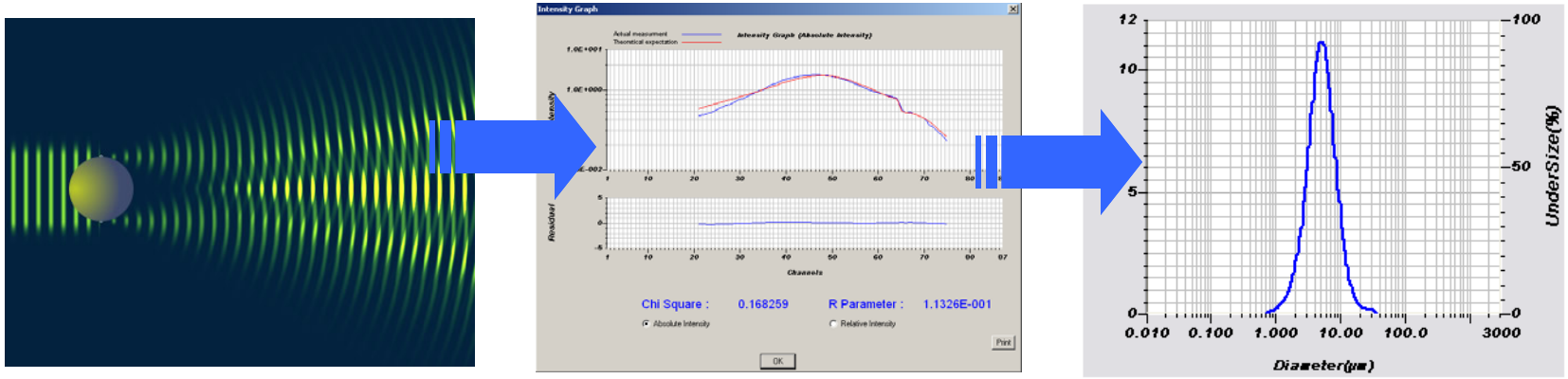


LA-960 Method Expert



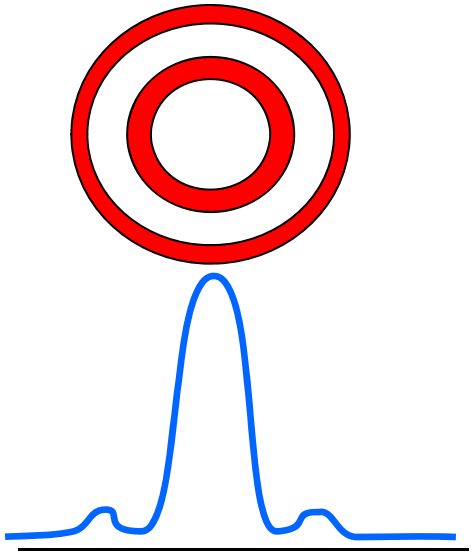
Intensity Graph

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

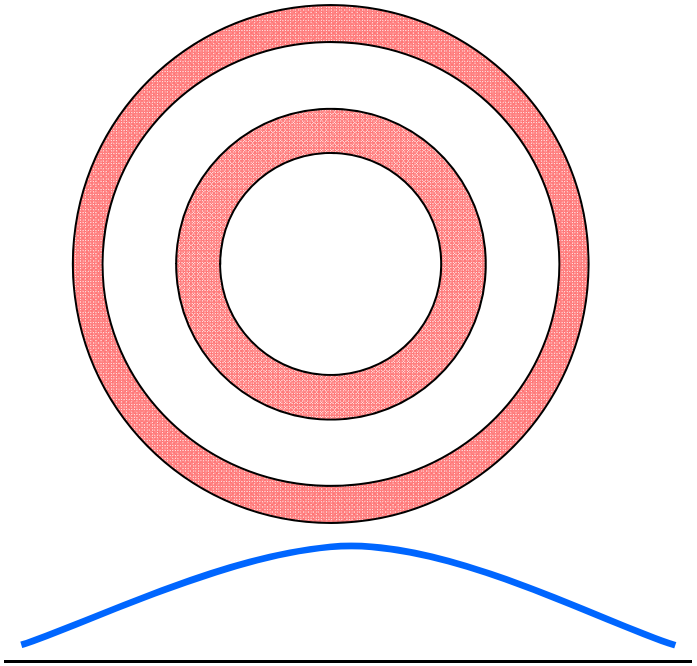


Size affects intensity

- LARGE PARTICLE:
  - Low angle scatter
  - Large signal



Narrow Pattern - High intensity

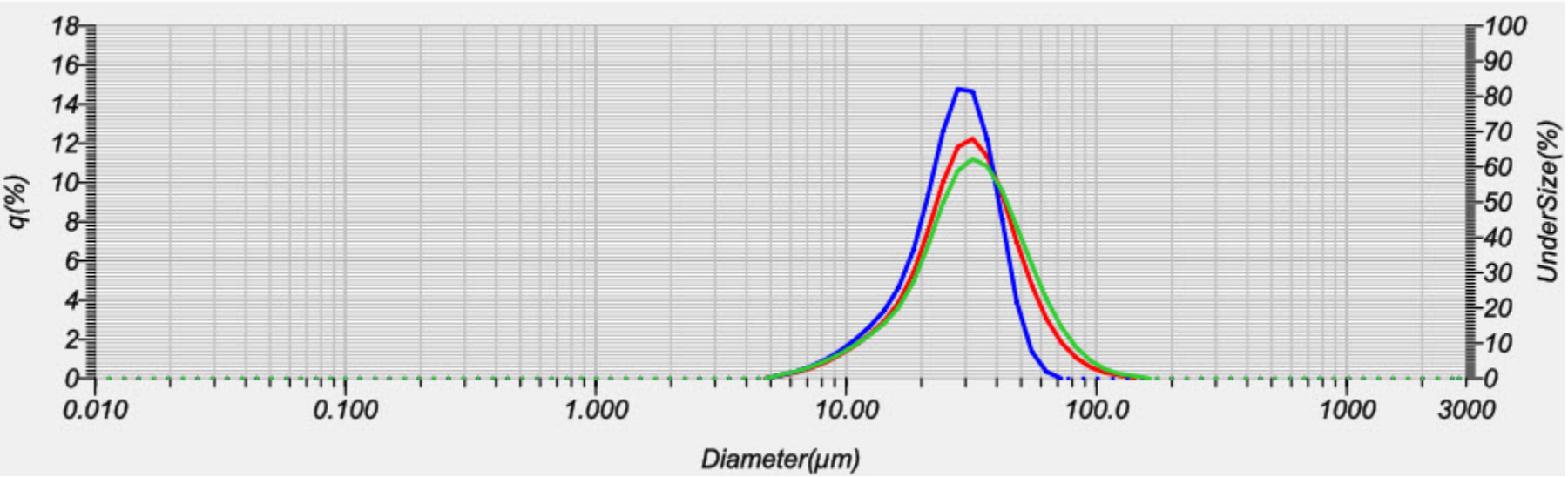


Wide Pattern - Low intensity

- SMALL PARTICLE:
  - High Angle Scatter
  - Small Signal

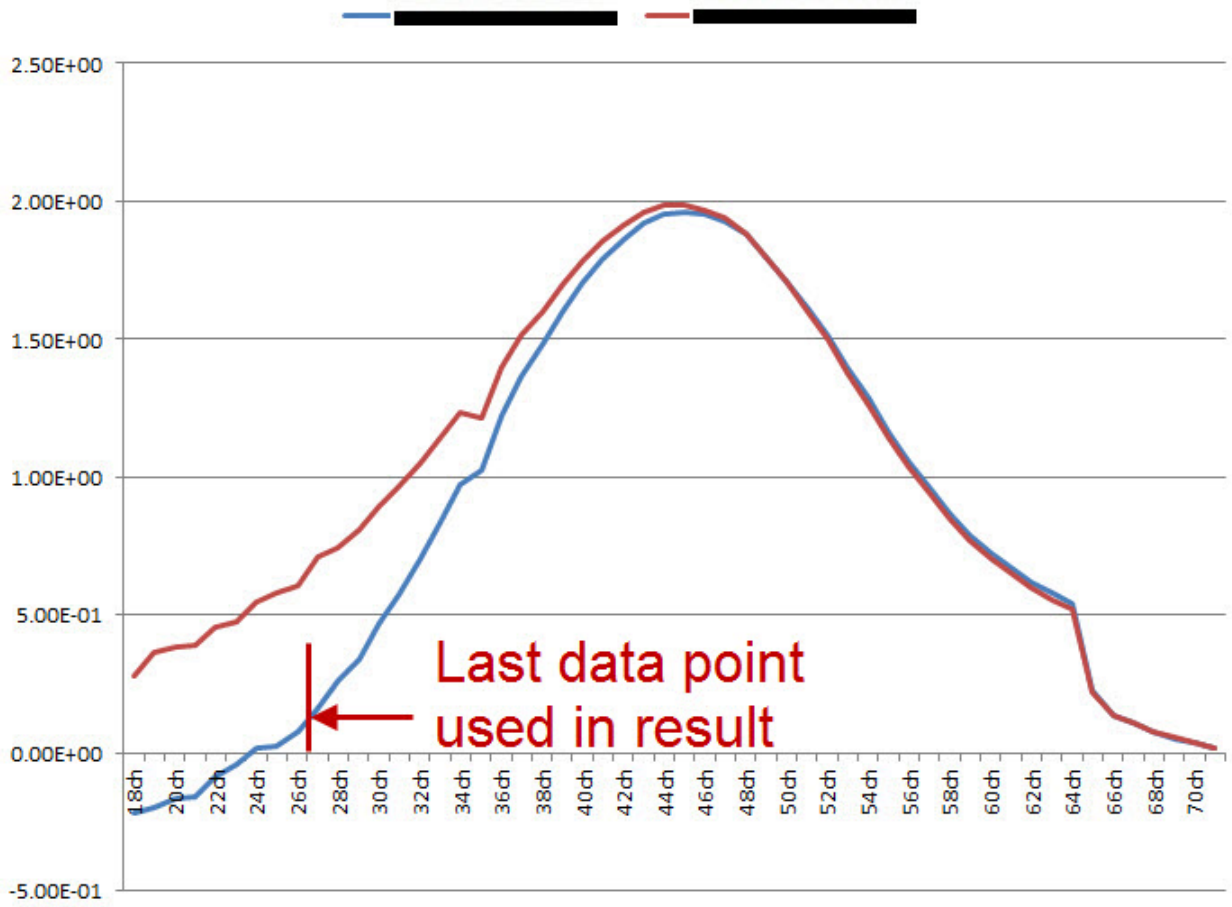
Intensity Graph

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



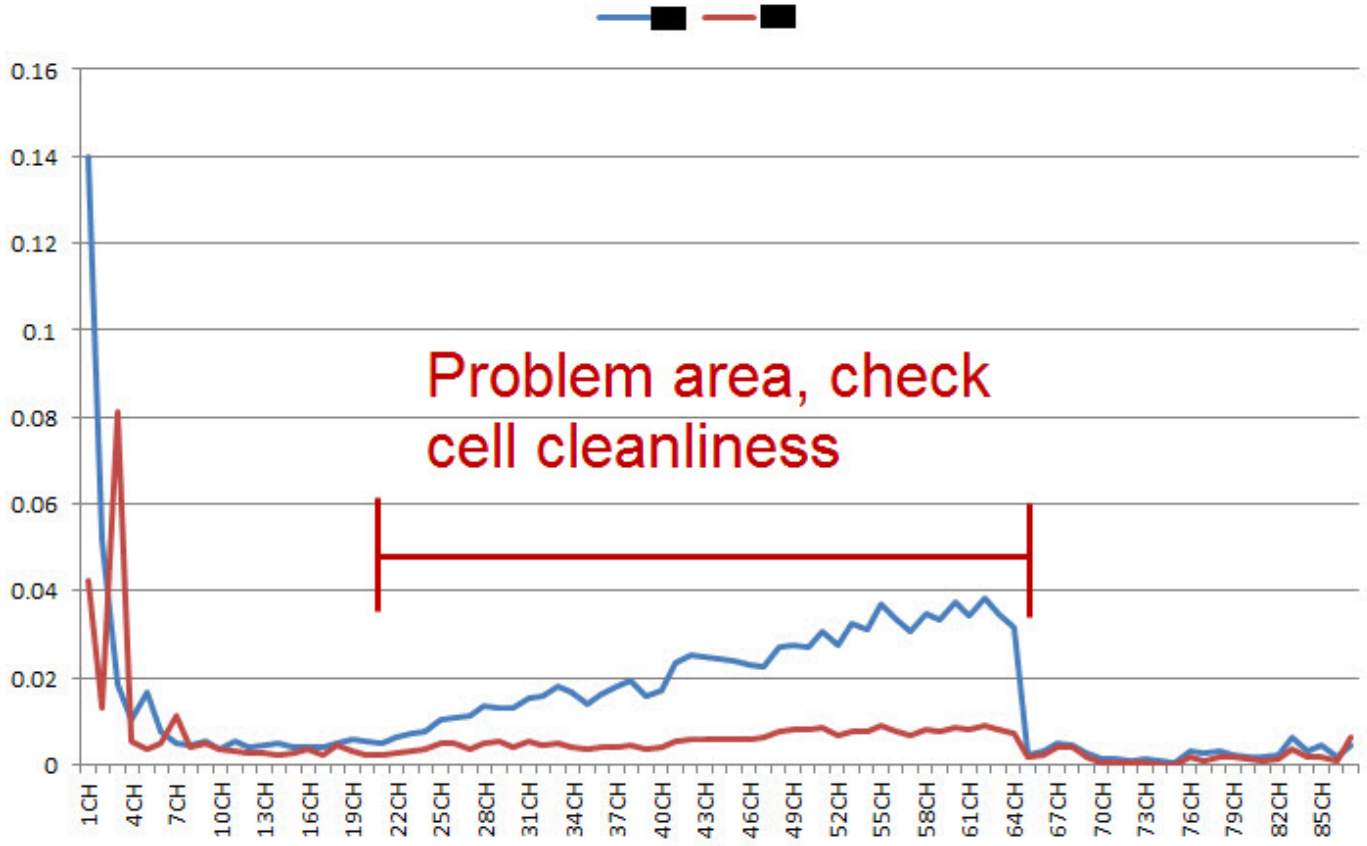
Intensity Graph

- Pull up the tool and compare



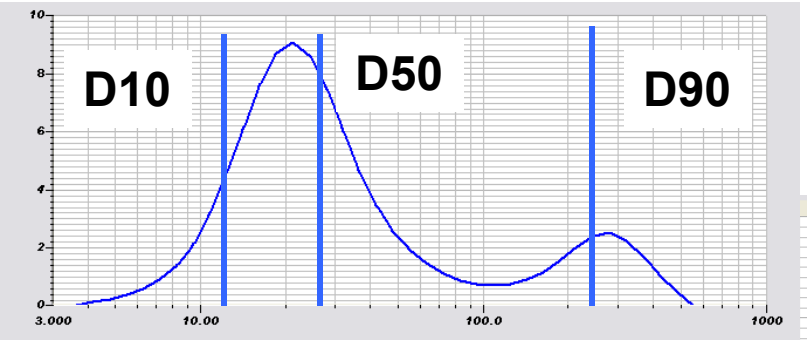
Intensity Graph

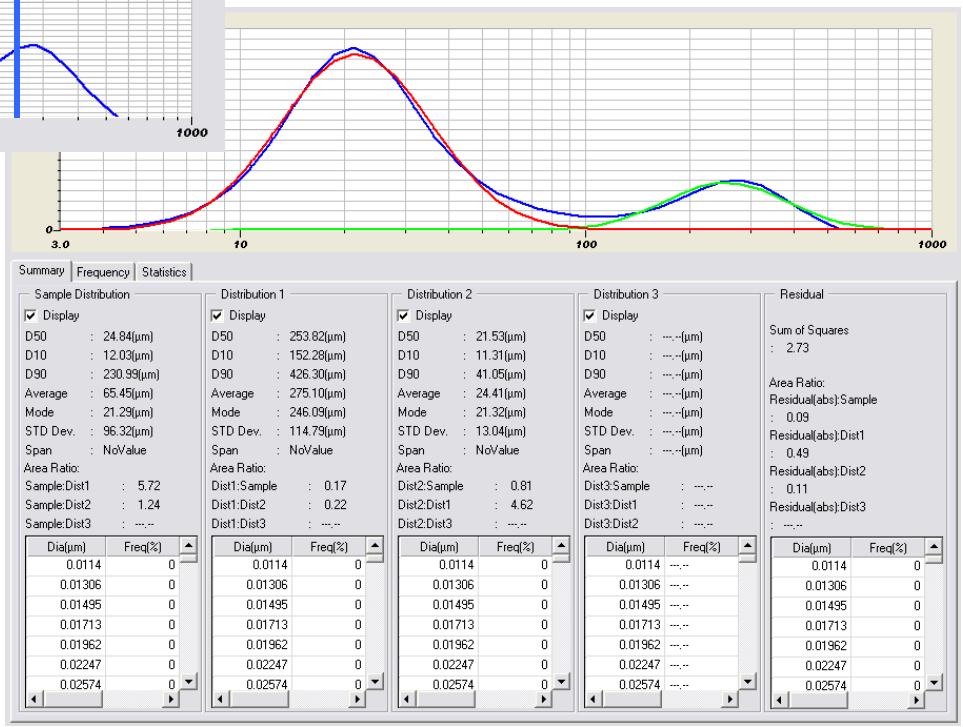
- Need to explain difference in scattering
- Try other tools, i.e. Blank Check



Multi-modal distributions

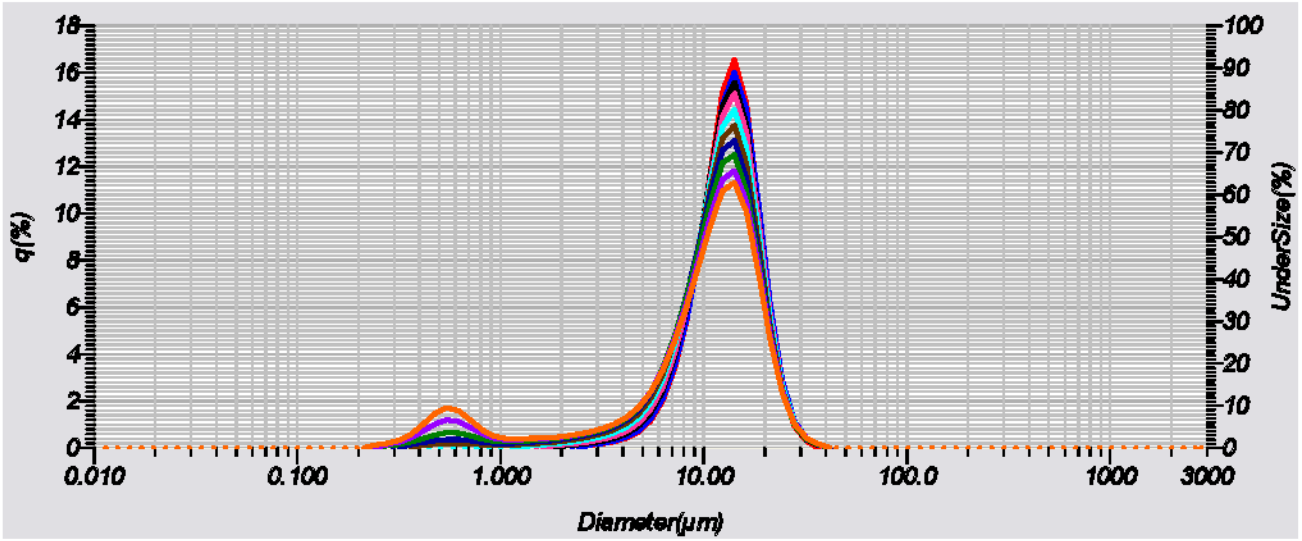
Multiple peaks can be better described individually





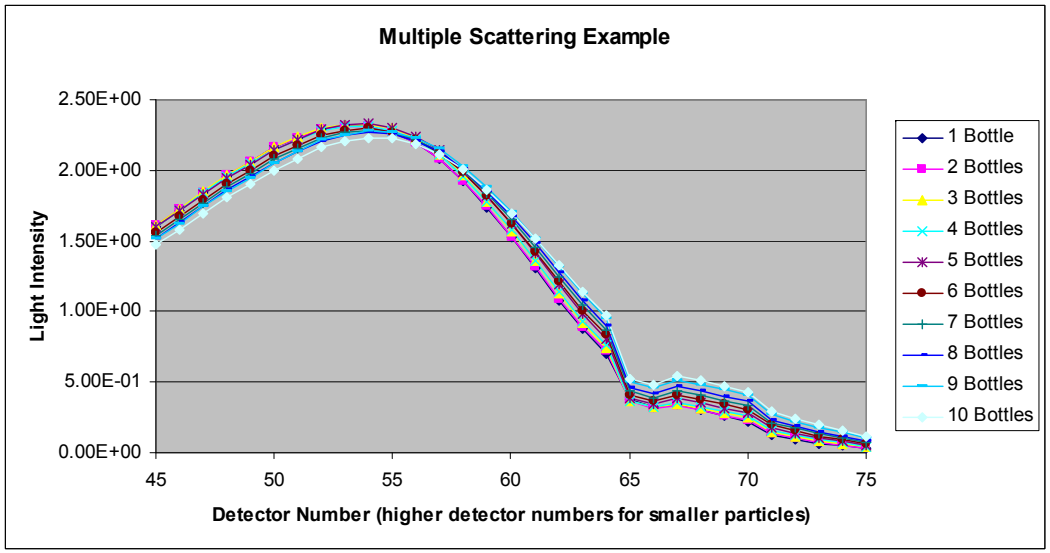
Multiple scattering

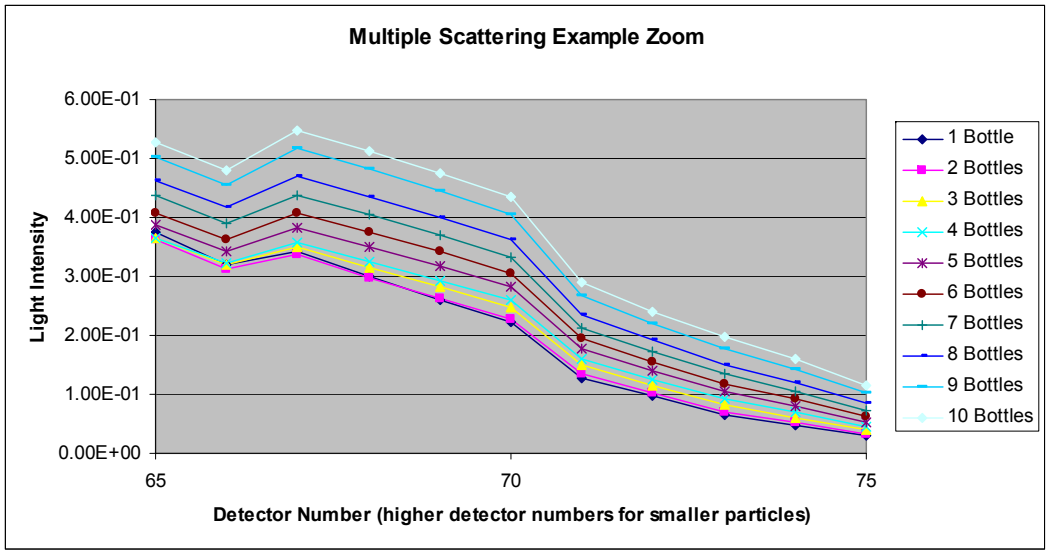
Watch for finer “particles” appearing with increasing concentration



Graph Type	Test or Assay. Number	Transmittance(R)	Transmittance(B)	Verification
	1 bottle	85.4(%)	85.2(%)	1.OK 2.OK 3.OK
	2 bottles	70.7(%)	71.8(%)	1.OK 2.OK 3.OK
	3 bottles	58.1(%)	60.2(%)	1.NG 2.OK 3.OK
	4 bottles	48.1(%)	51.9(%)	1.NG 2.OK 3.OK
	5 bottles	39.4(%)	44.7(%)	1.NG 2.OK 3.OK
	6 bottles	32.4(%)	38.6(%)	1.NG 2.OK 3.OK
	7 bottles	27.7(%)	34.1(%)	1.NG 2.NG 3.OK
	8 bottles	23.4(%)	29.9(%)	1.NG 2.NG 3.OK
	9 bottles	18.9(%)	25.9(%)	1.NG 2.NG 3.OK
	10 bottles	15.9(%)	22.7(%)	1.NG 2.NG 3.OK

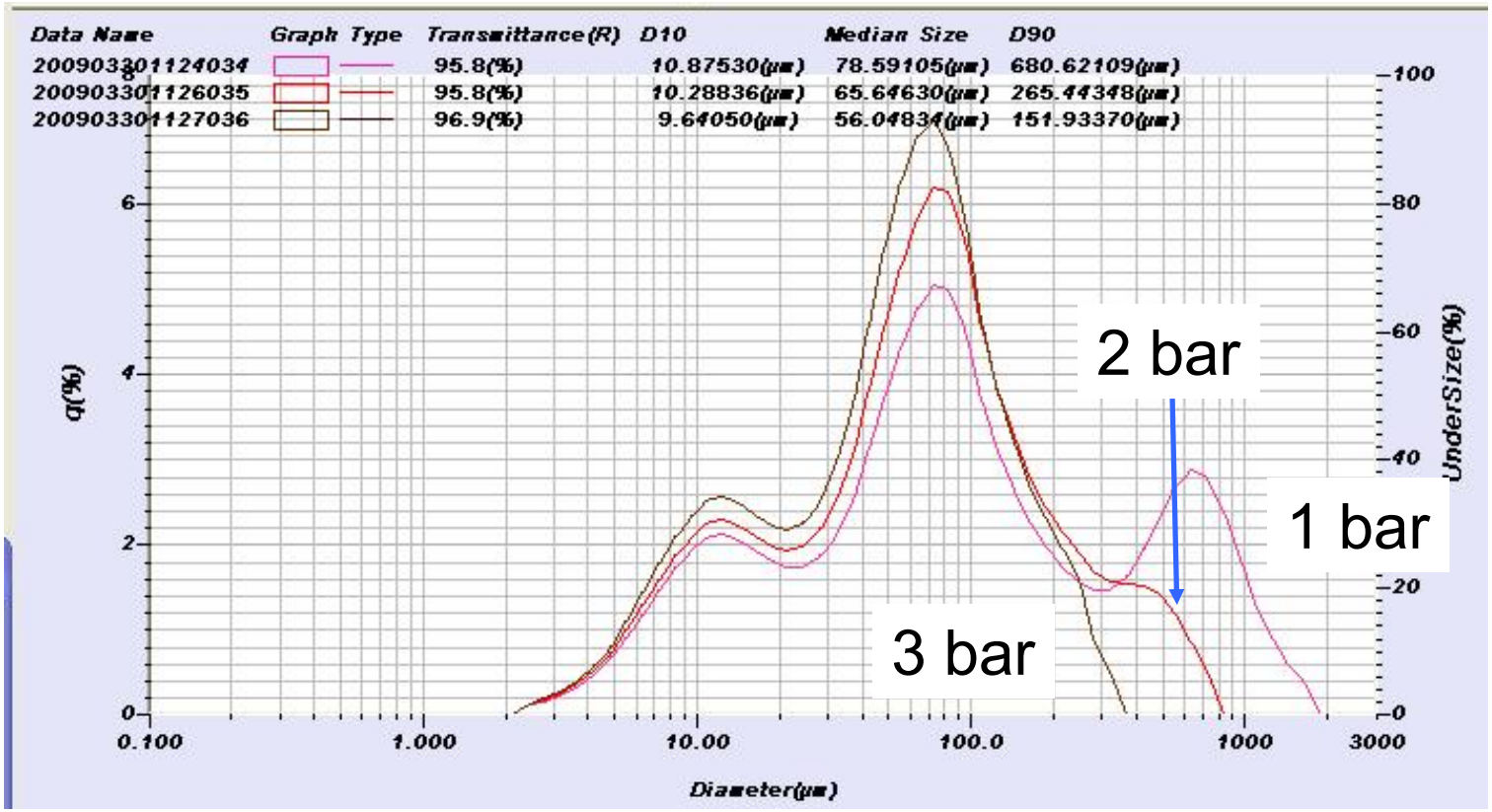
Multiple scattering





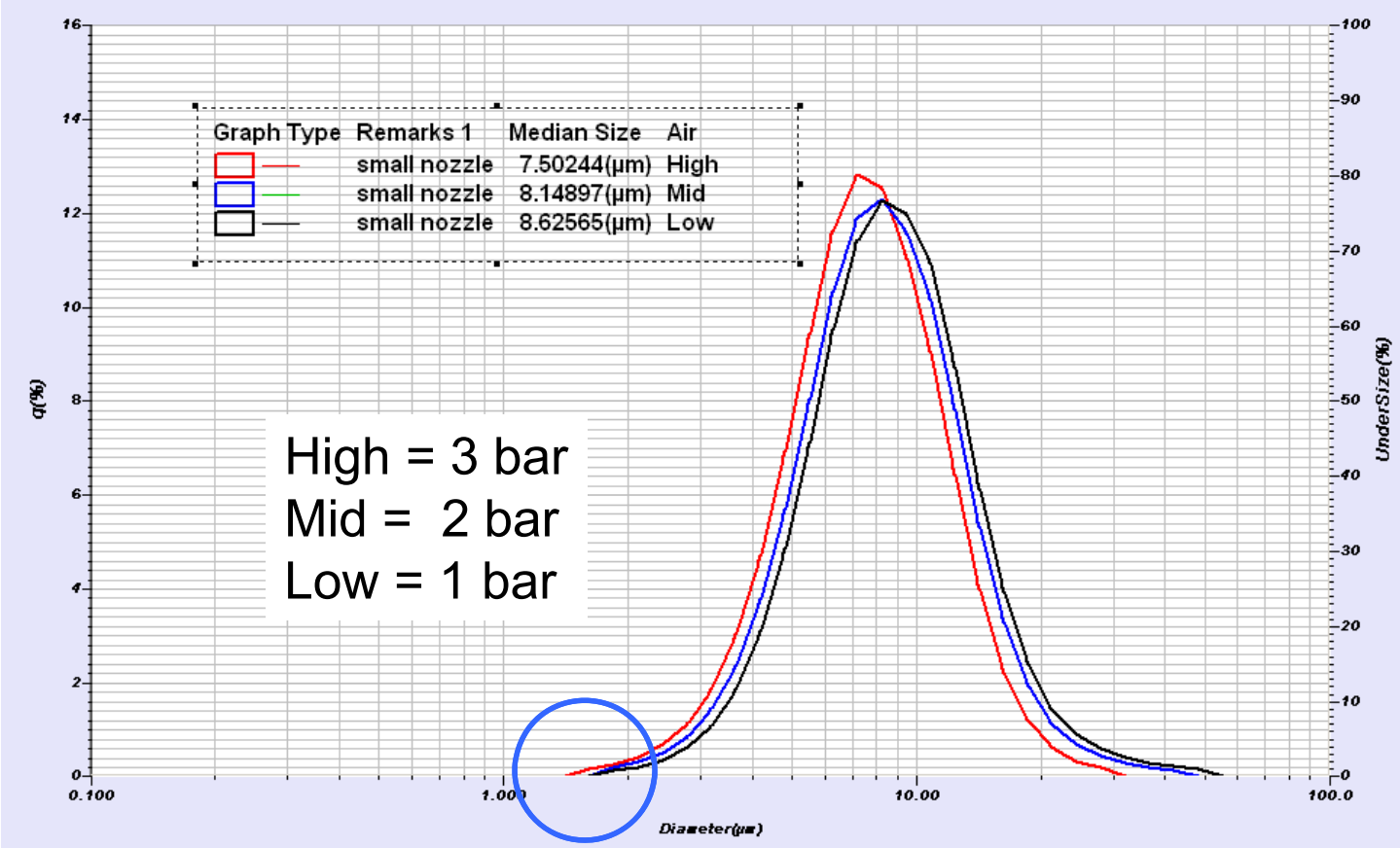
Dispersing agglomerates

Watch for no change in coarsest particles with changing energy



Breaking particles

Watch for finer particles being created with increasing energy



HORIBA

Thank you

ありがとうございました

Cảm ơn

Dziękuję

ขอบคุณครับ

谢谢

Gracias

Grazie

اشْكُر

Σας ευχαριστούμε

धन्यवाद

நன்றி

Tacka

Danke

Merci

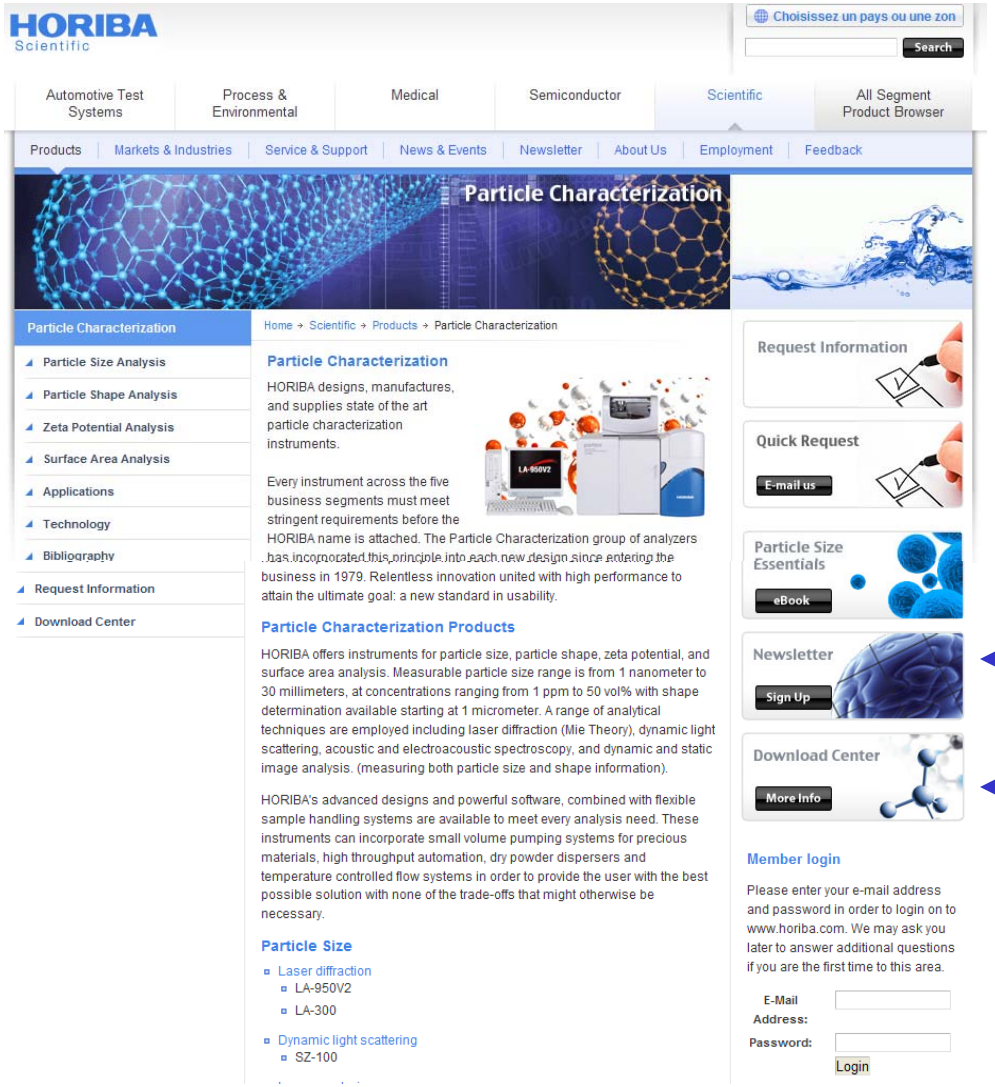
Obrigado

감사합니다

Большое спасибо

www.horiba.com/particle

HORIBA



Ian Treviranus
Product Line Manager
ian.treviranus@horiba.com

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labinfo@horiba.com

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