HORIBA Scientific
Particle Characterization

Laser Diffraction Academy: Choosing the Best Dispersion Tools for Your Samples

Jeffrey Bodycomb, Ph.D.

April 27, 2023

Overview

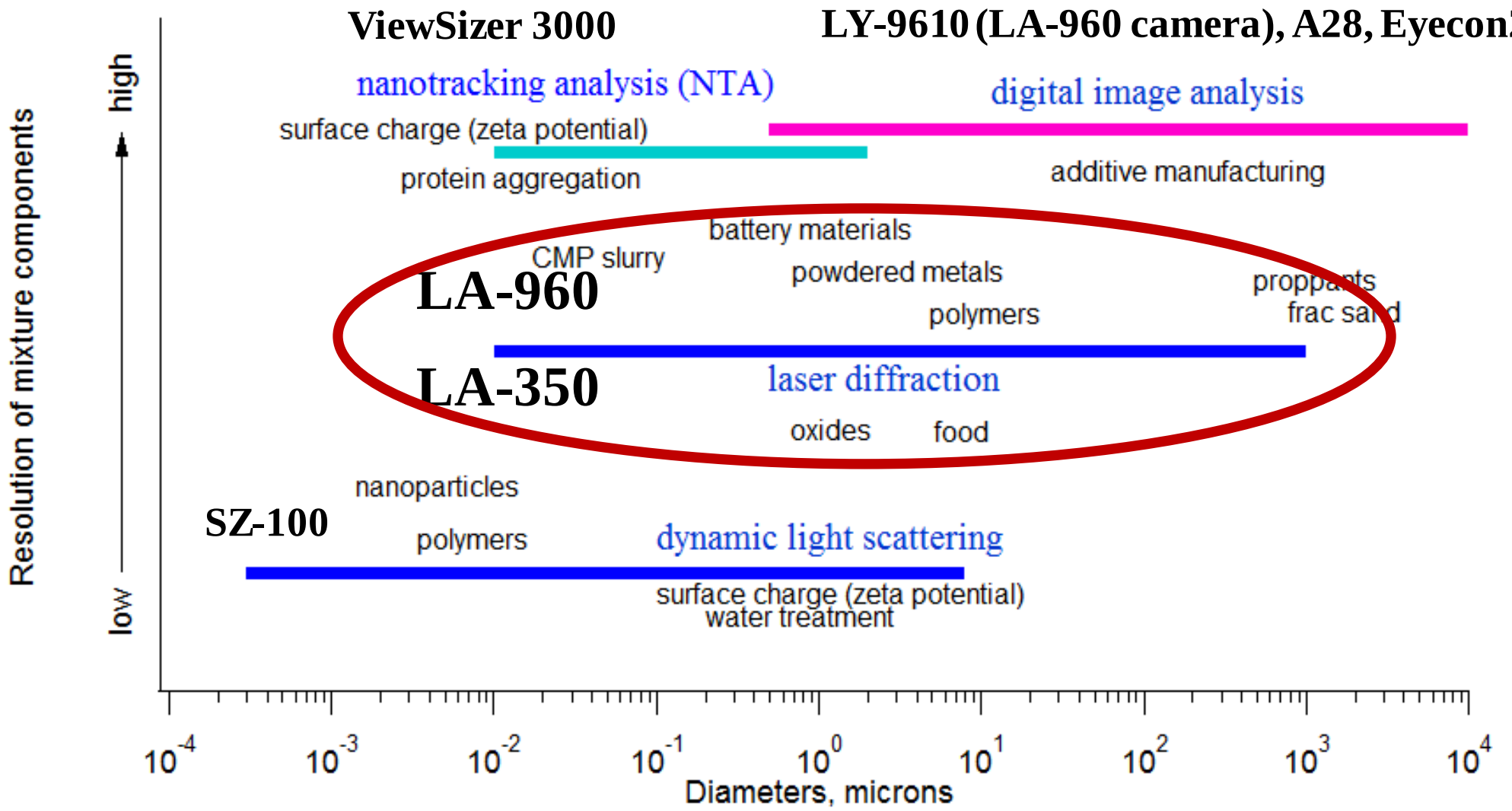
Introduction

Options for liquid dispersions
largest to smallest volumes

Dry powders

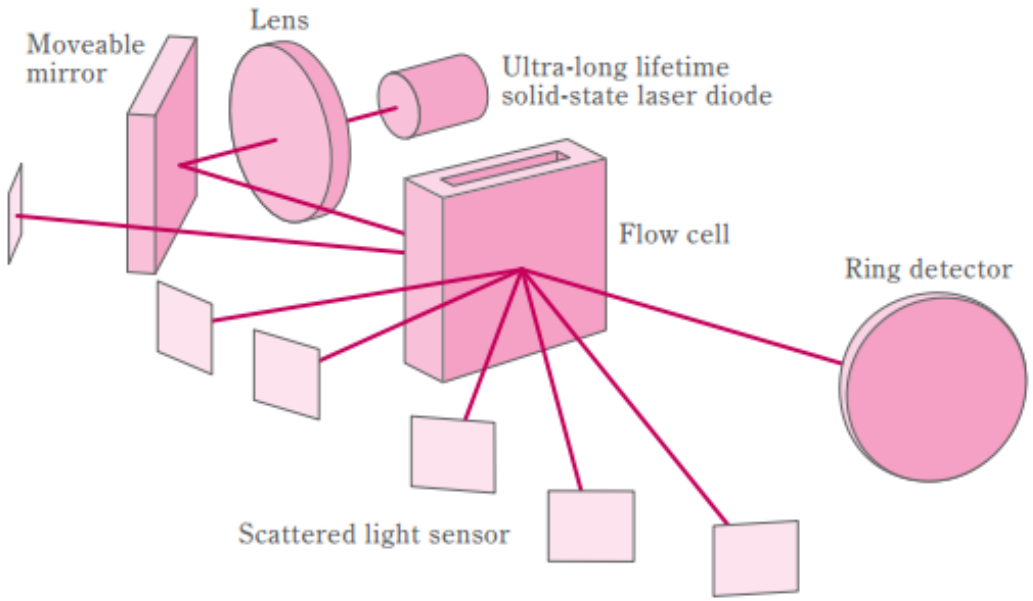
Concluding comments

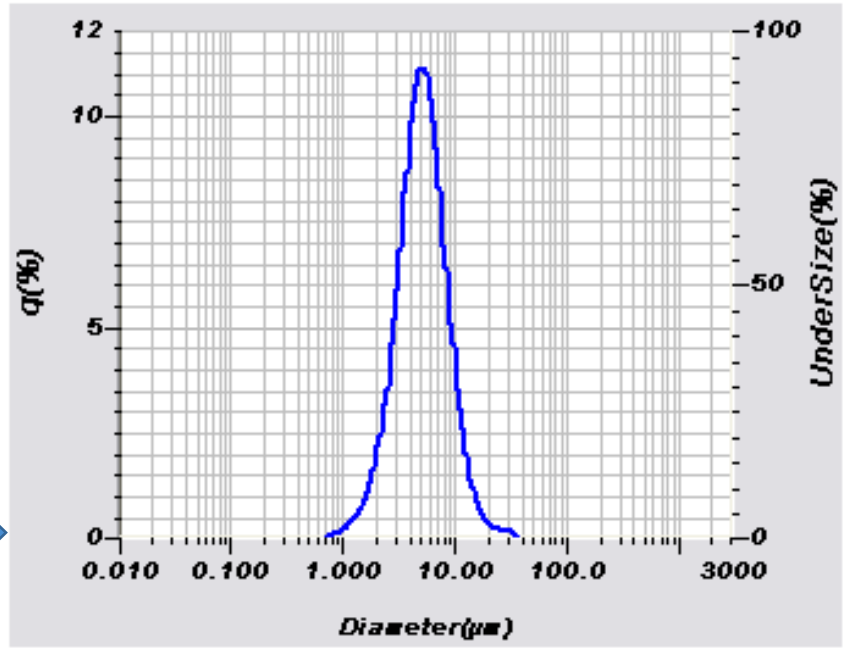
Perspective



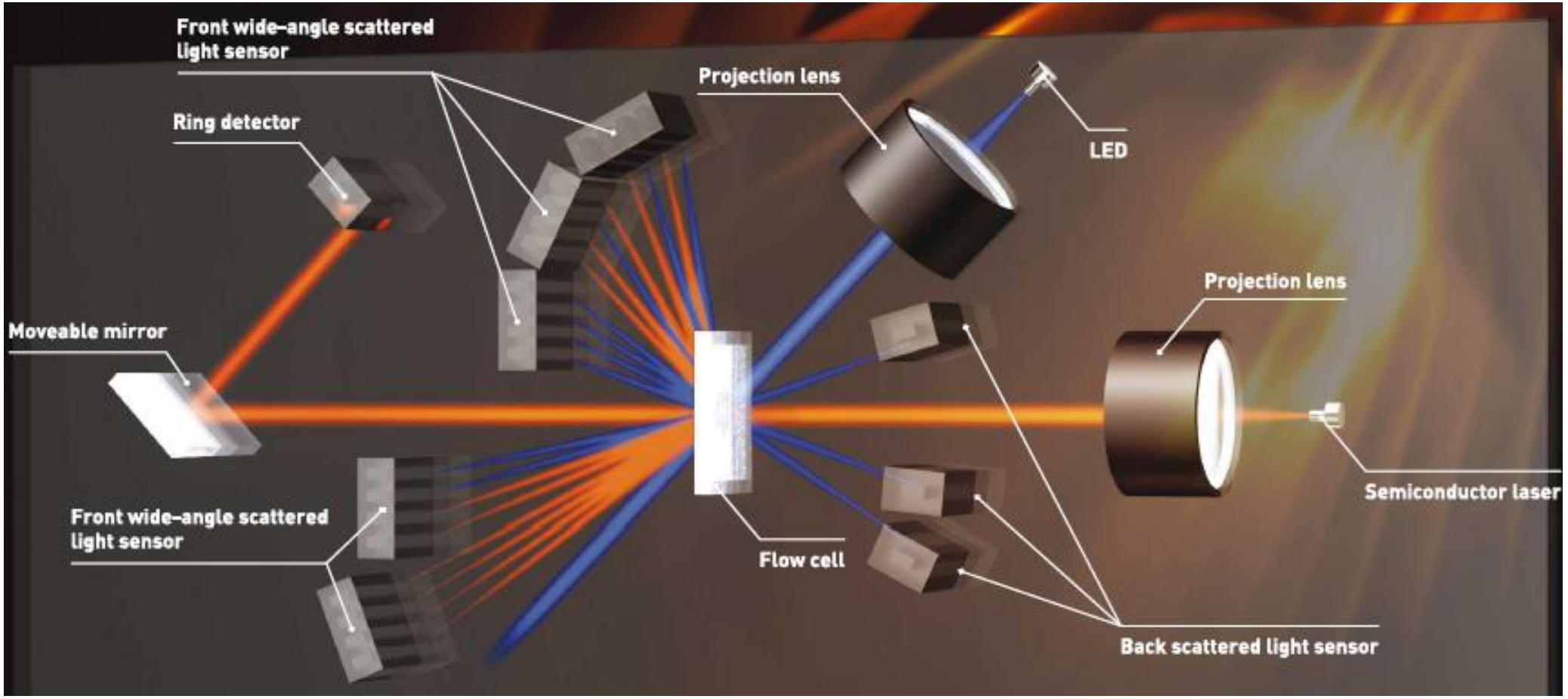
Core principle

Investigate a particle sample with light and determine size distribution.



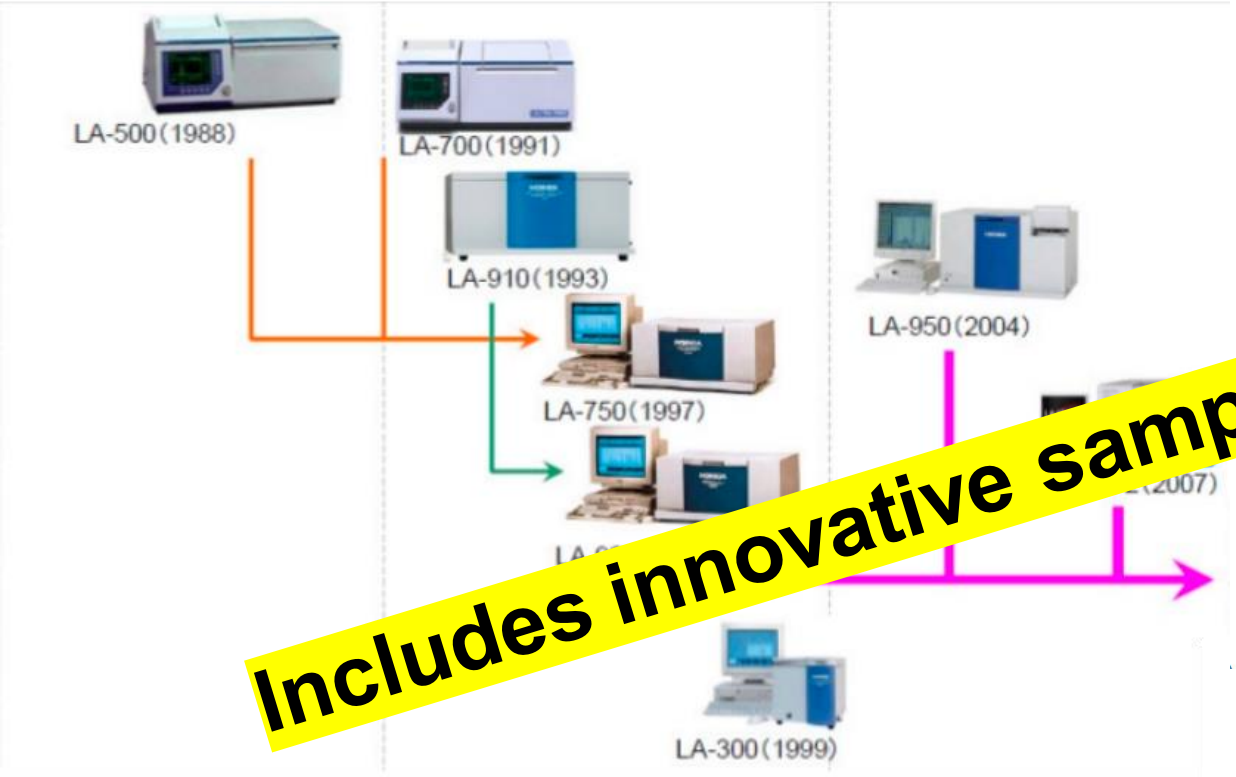


LA-960 optics



Angular range: ~0.006 degrees to ~165 degrees

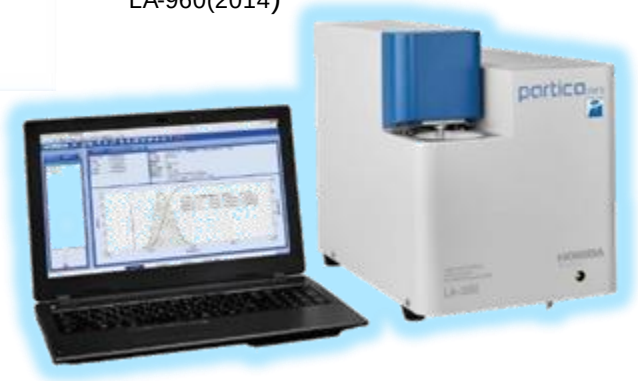
History of successful innovation



Includes innovative sample handling.

LA-960(2014)

LA-960V2



LA-350

Sample handling decision drivers

Particle size (affects settling, dispersion)

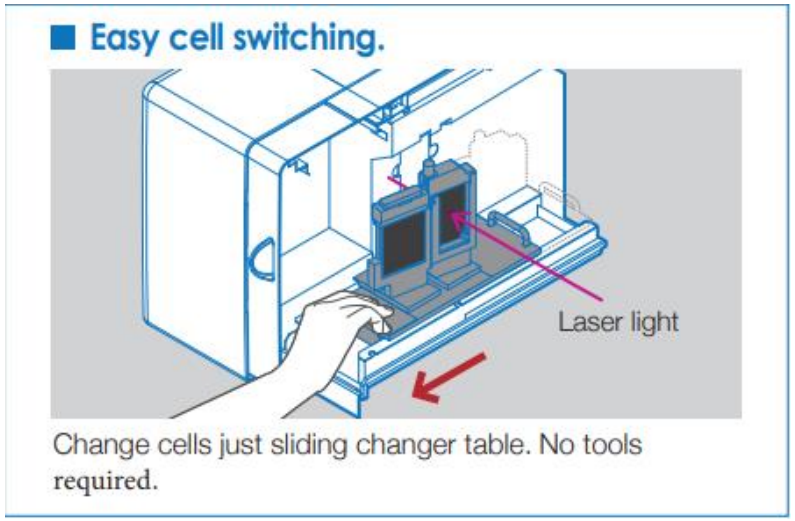
Distribution width

Available sample quantity

Sample form (dry powder vs liquid dispersion)

Dispersant type(s)

Accessories for wet analysis



Flow Cell

Volume (mL)	180 – 250
Range (µm)	0.01 – 3000

Mini flow

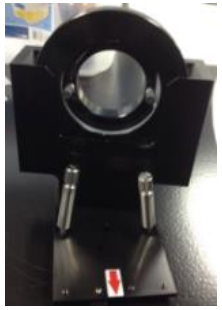
Volume (mL)	35
Range (µm)	0.01 – 1,000

Fraction Cell

Volume (mL)	5 – 15
Range (µm)	0.01 – 3000

Paste Cell

Volume (mL)	~0.3
Range (µm)	0.01 – ~500



HL Cell

Volume (mL)	0.5 – 2 mL
Range (µm)	0.01 – 500

LiterFlow

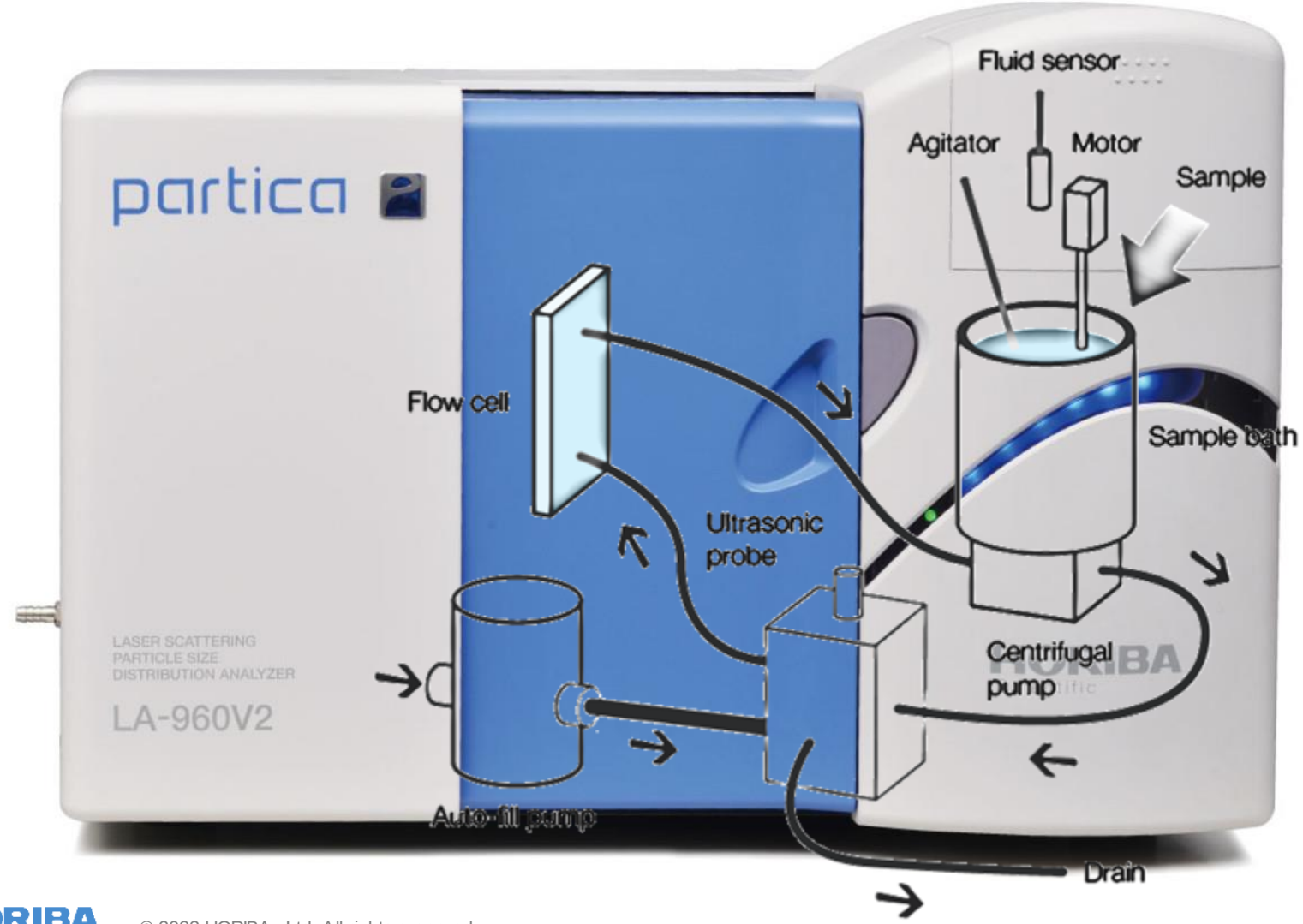
Volume of 1 Liter

Even larger volume for wide size distributions.

This is rarely used. One application is soil samples.

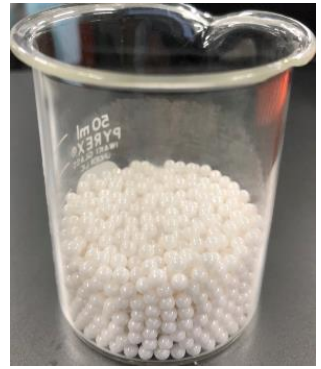
Standard circulation (Aqua/Solvo flow)

180 – 250 mL volume



Circulate dense 3 mm particles.

Why? Because we can…



※Ceramic Zirconia
3mmΦ

Standard Circulation

180 – 250 mL volume

needs more sample

needs more liquid (solvents can get expensive, water is usually inexpensive)

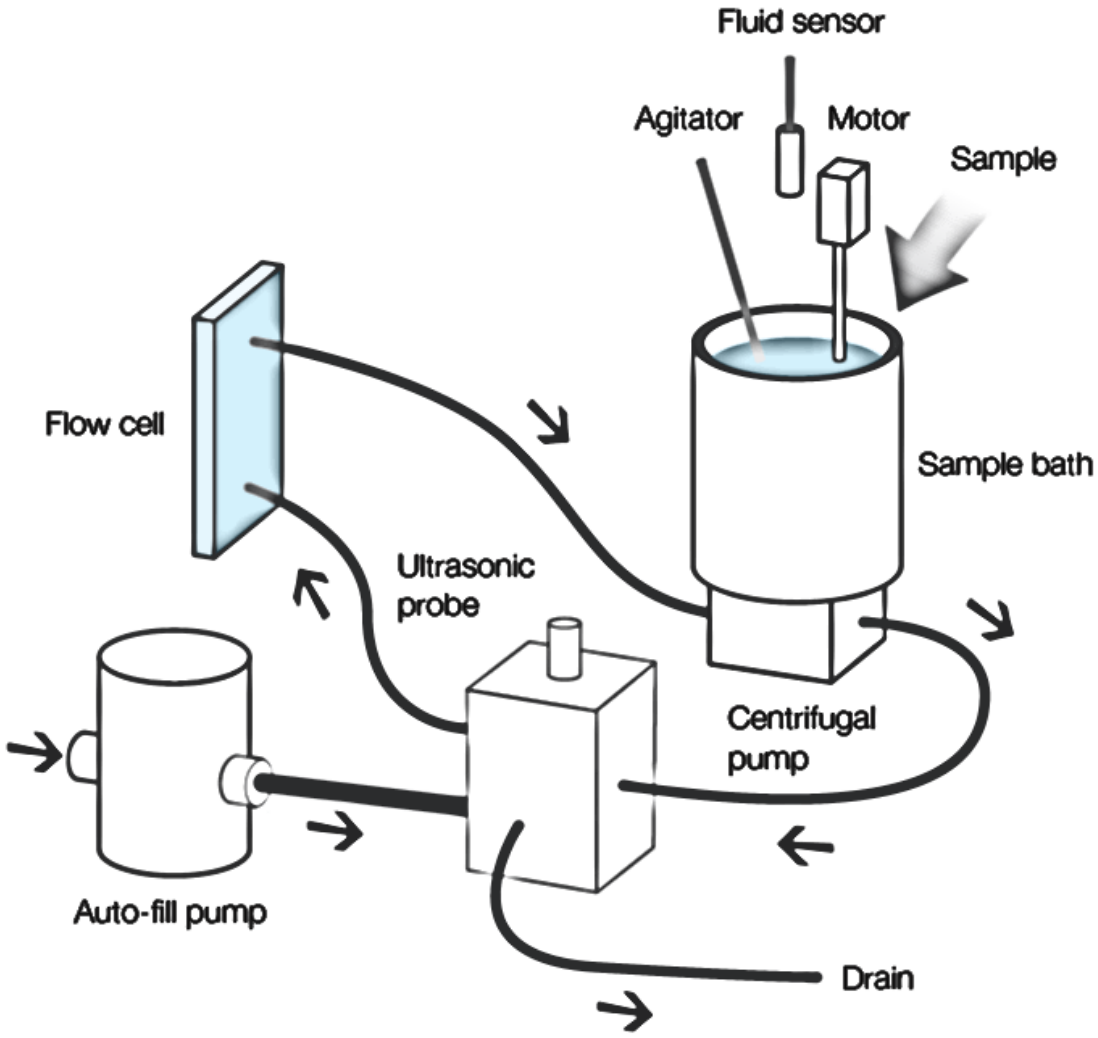
Easy to add sample and not add too much since it takes a lot of sample to change concentration

Easy to clean, just press the rinse button!

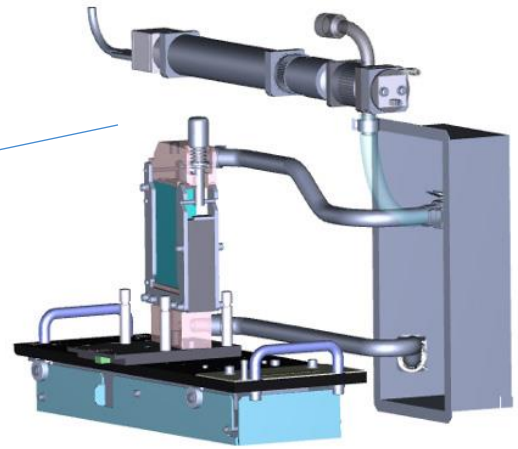
Lower cost

Can be used with imaging accessory, slurry sampler

Solvent resistant version also available and required if using organic liquids

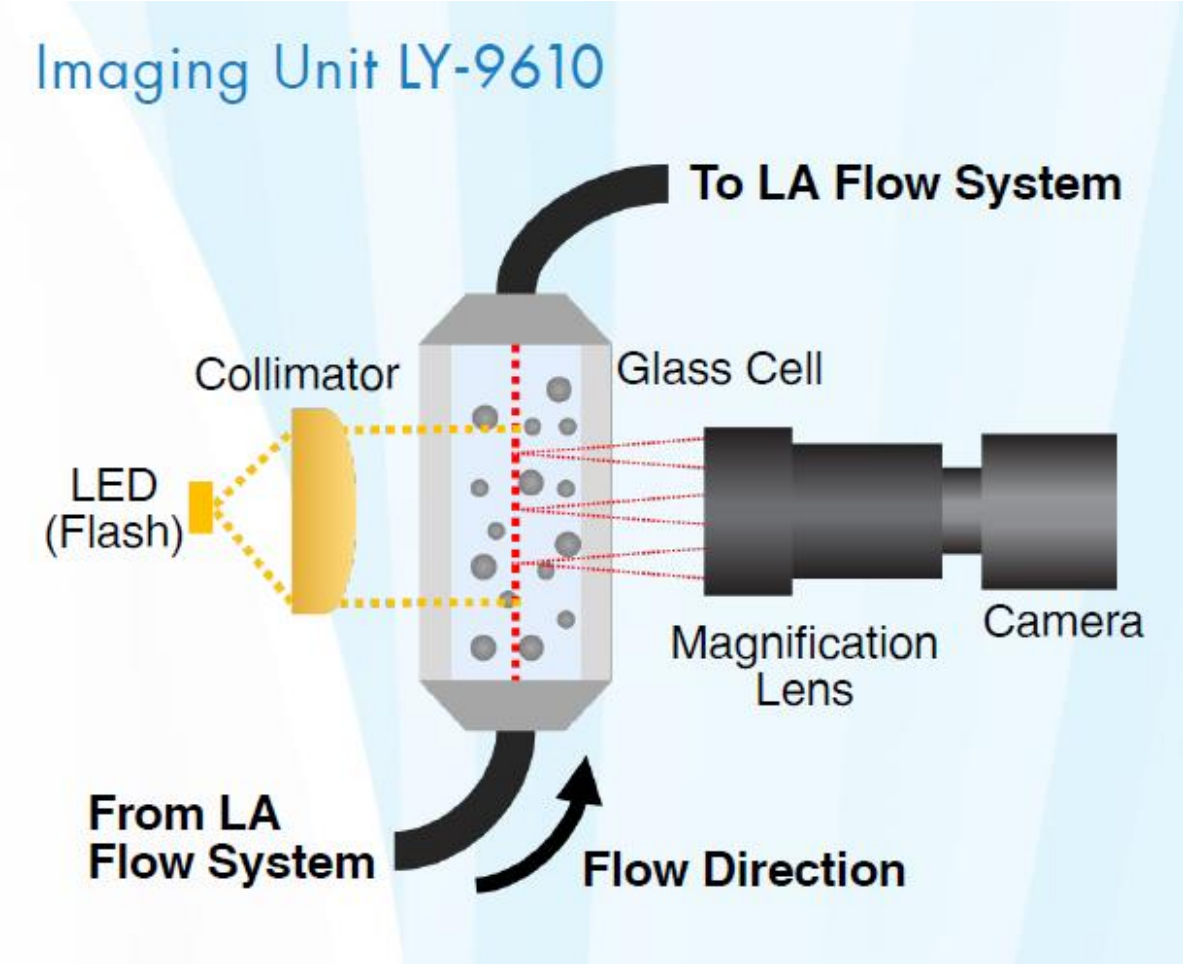


Imaging option



Integrated Module!

Imaging



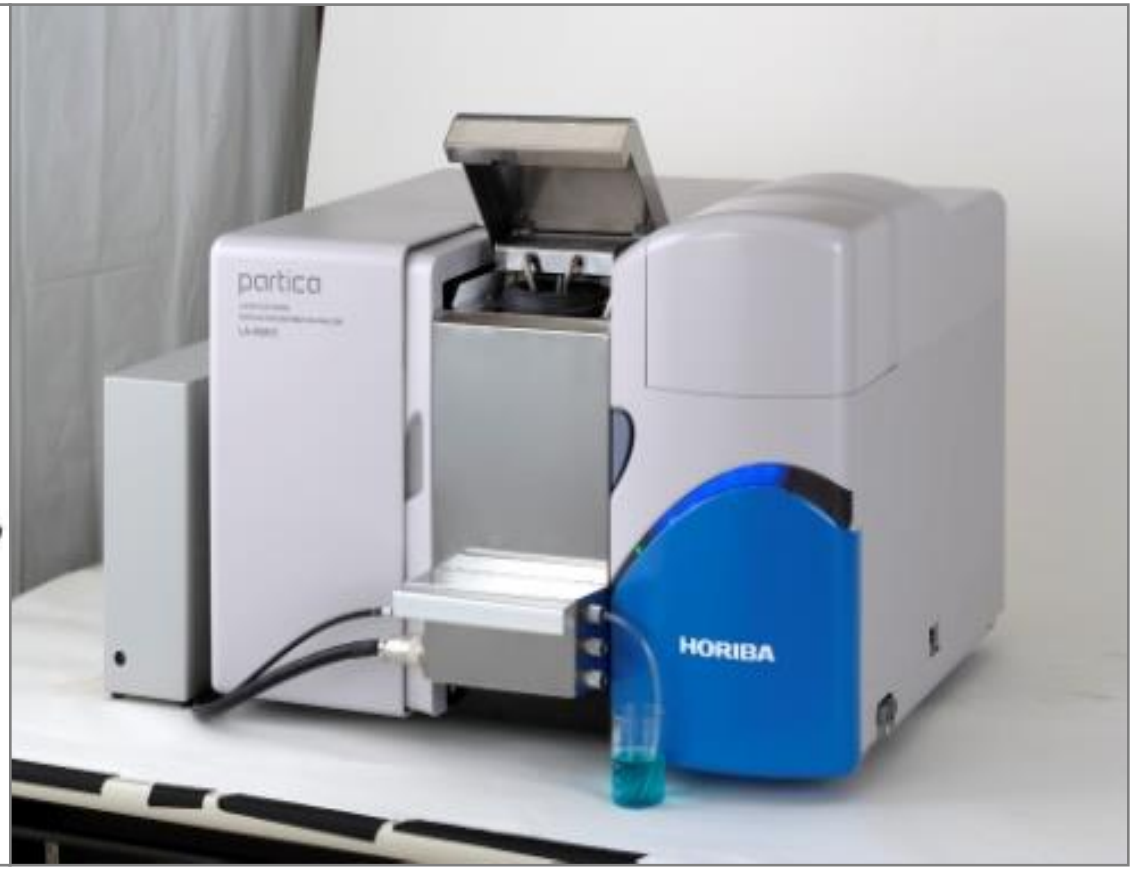
Size Range: 9-1000 microns

Pixel Size: 0.73 microns

Autosamplers for Aqua/Solvoflow

MiniFlow

35 ~ 50 mL volume



MiniFlow

35 ~ 50 mL volume –Smaller but still has on board fill pump, ultrasound

Solvent resistant. Solvent is expensive and the miniflow is a good choice if using organic liquids.

Can set up with a large volume aqueous and miniflow for solvent. No need for arduous cleaning.

Fraction cell

5 ~ 15 mL volume (30x less material)

Glass cuvette

Mixing is only a stir bar

No mixing for the smallest volume cuvettes

Reproducibility

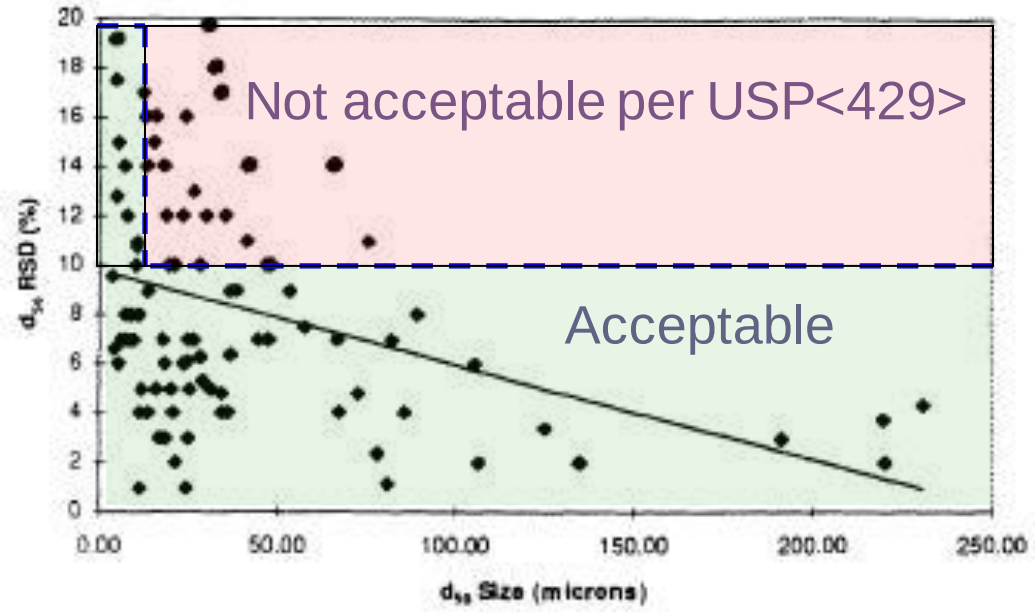
58 methods

Image analysis for morphology

Laser diffraction for PSD

If RSD for d50 < 20%, then acceptable for QC environment

Note: RSD increases with decreasing size

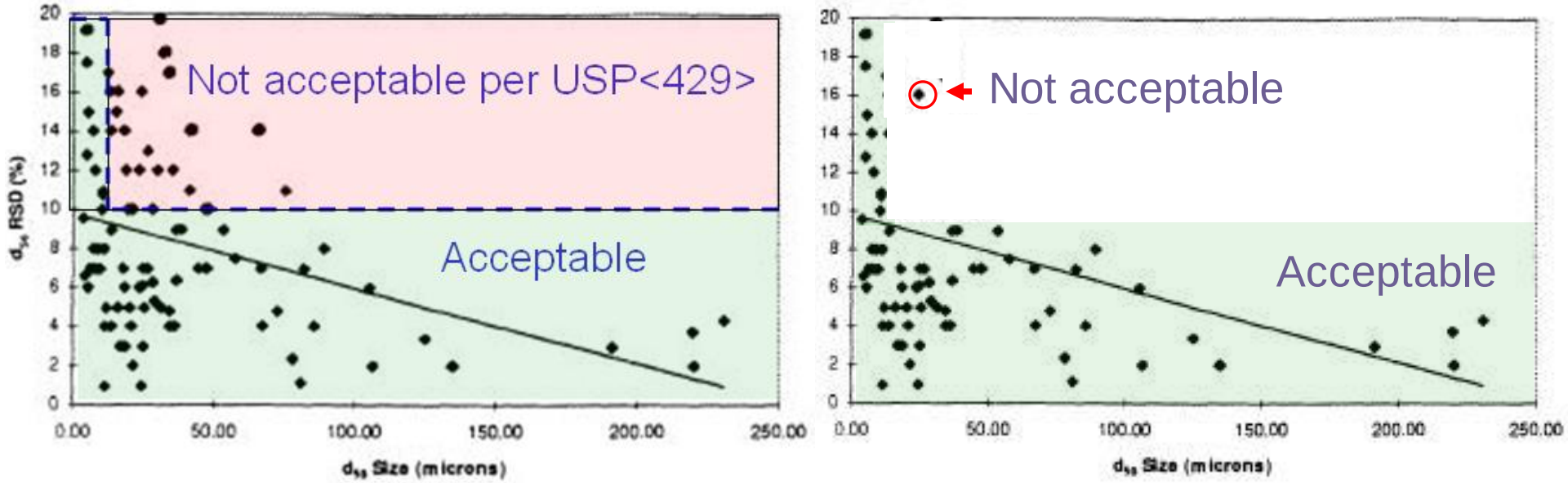


*Barber, Keuter, and Kravig, A Logical Stepwise Approach to Laser Diffraction Particle Size Distribution Analysis Methods Development and Validation Pharmaceutical Development and Technology, 3(2), 153-161 (1998)

HORIBA Scientific

Sampler Selection

Remove points from not acceptable region using Fraction Cell



*Barber, Keuter, and Kravig, A Logical Stepwise Approach to Laser Diffraction Particle Size Distribution Analysis Methods Development and Validation Pharmaceutical Development and Technology, 3(2), 153-161 (1998)

Fraction cell

5 ~ 15 mL volume (30x less material)

Glass cuvette

Mixing is only a stir bar
No mixing for the smallest volume

Upper size limit depends on density, but we avoid this option if sizes are over 10 microns.

High concentration cells

0.5 - 2 mL volume

Setup and cleaning is difficult here.

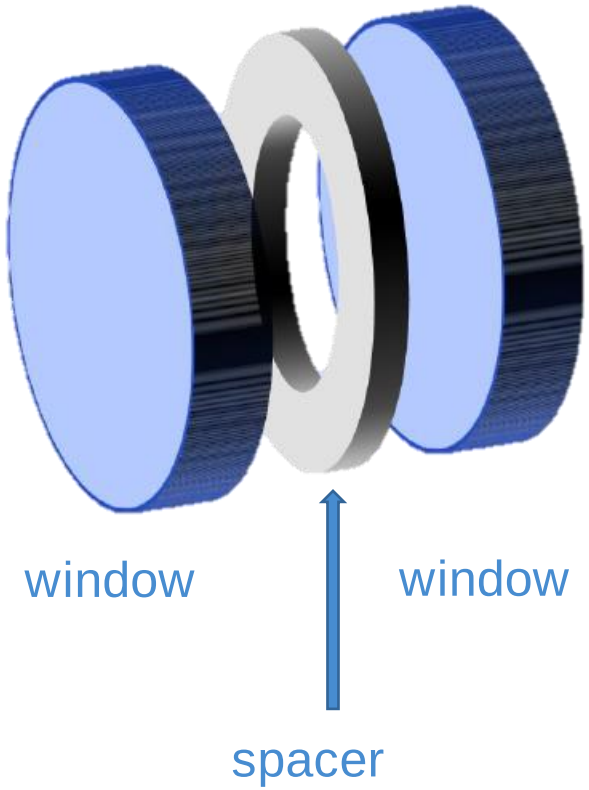
More dexterity and less button pressing.

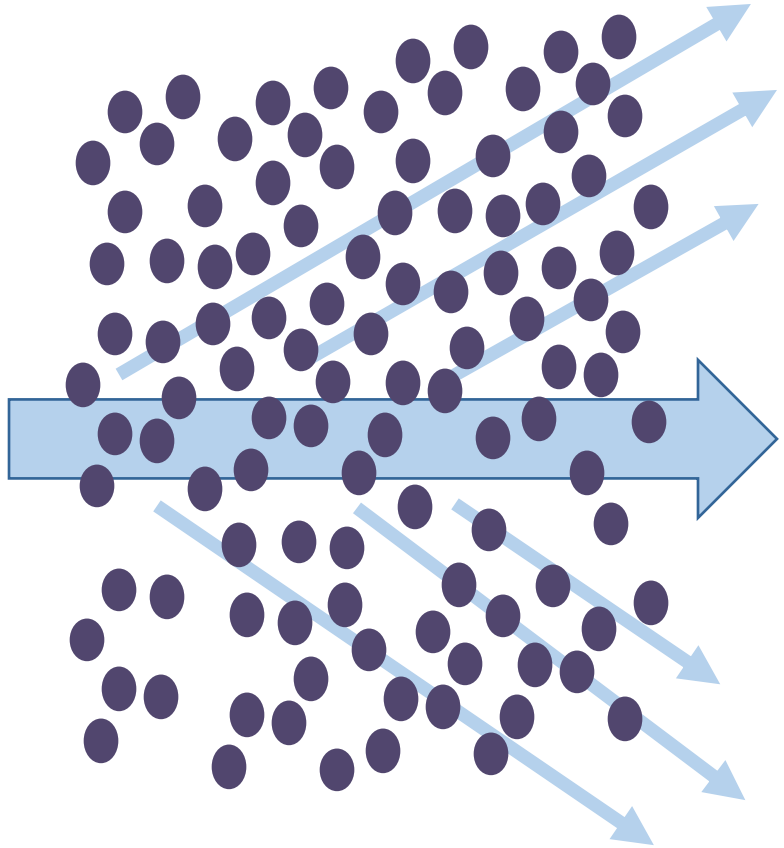
Particle Sciences (Bethlehem, PA):
“Bubbles, bubbles, bubbles!!! Very important to minimize”

Generally only if nothing else will do.

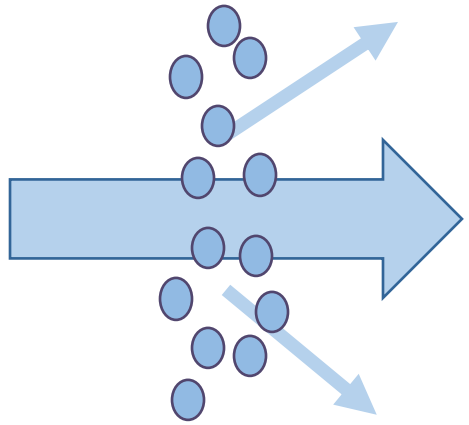
Just a drop of sample is enough…

How?





Long path



Short path

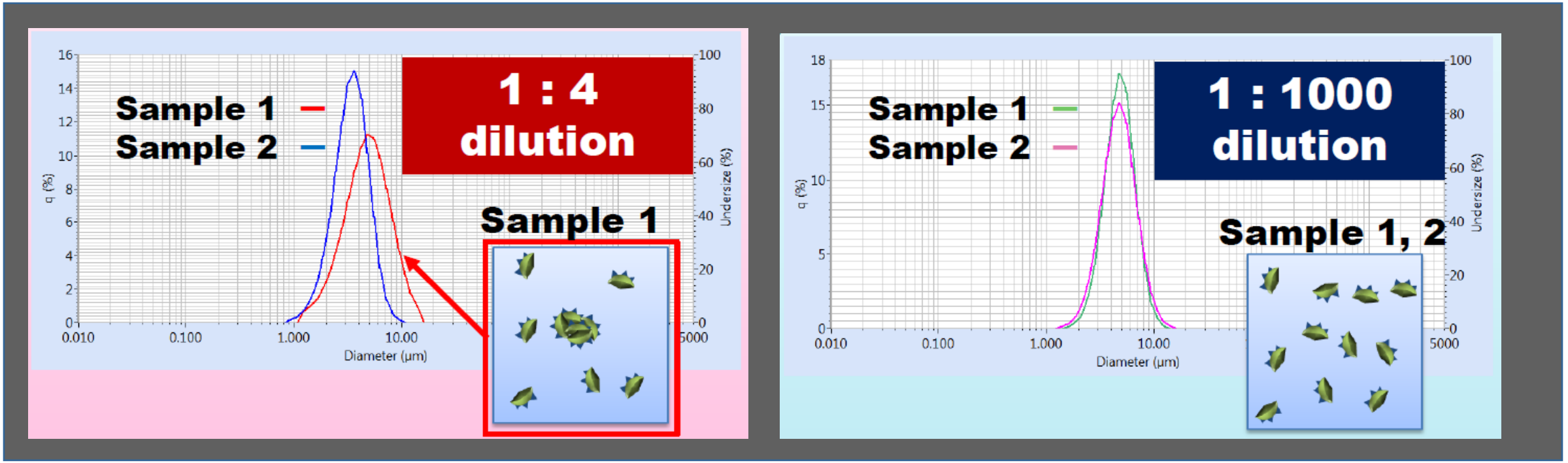
Narrow spacer means short optical path length.

Mechanics of use

Why?

Battery Electrode:

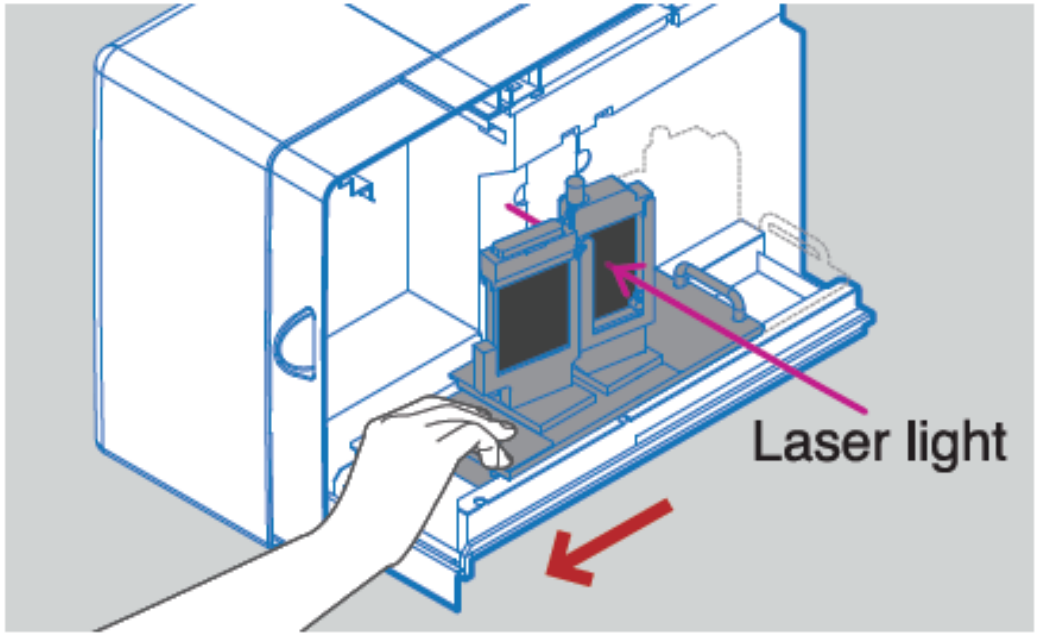
Sample 1 and 2 had different performance.



High dilution can suppress the interesting aggregation.

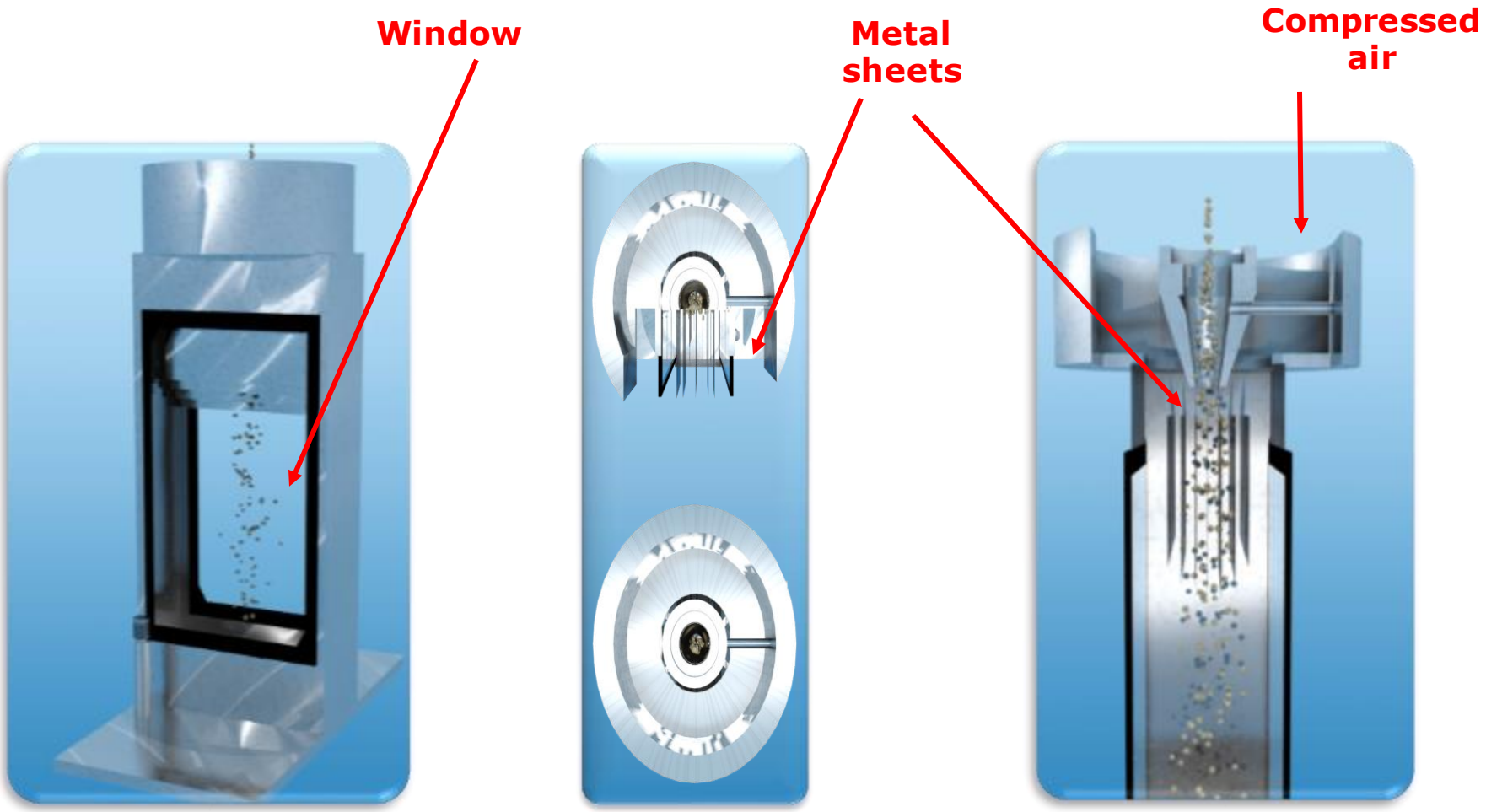
Switching cells

Flow cell	Nothing to do
Fraction cell	Minimum Steps! Remove tubes and just to slide the cell stage
High concentration cell	
Paste cell	
Dry cell	
Mini-flow unit	Remove imaging unit and pull out the stage, insert the Mini-flow unit.



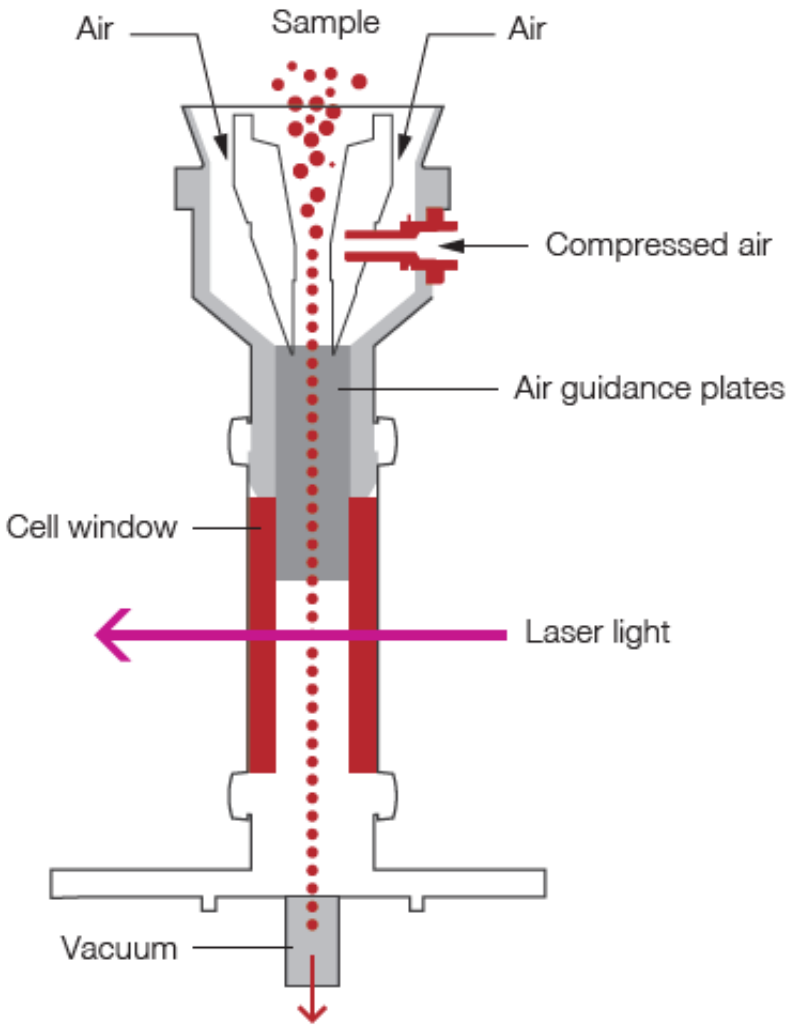
Also works when switching to dry….
….minimum steps, less work, fewer mistakes

Dry Dispersion



Dry powder feeder

- Direct flow of powder straight down
- Adjustable air pressure for dispersion.
- No impact surfaces means good dispersion w/o comminution.
- Feedback control gives great reproducibility.
- As little as 5 mg of sample.



HORIBA Scientific

Need a sample that flows OK

Really this is a tool for dry powders.

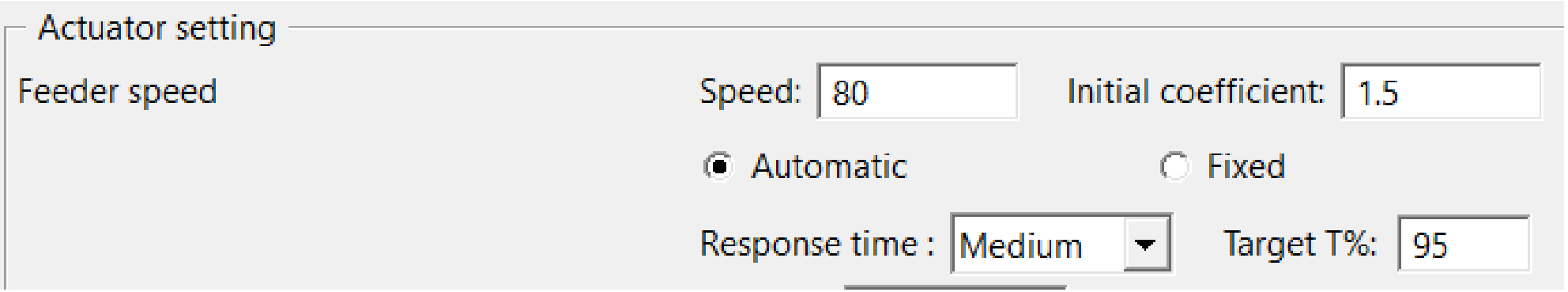
Air pressure can break up agglomerates but won't work for a sticky mess.

Particles under 1 micron are very difficult to separate due to surface effects. Therefore, the dry powder feeder is better for sizes over 1 micron.

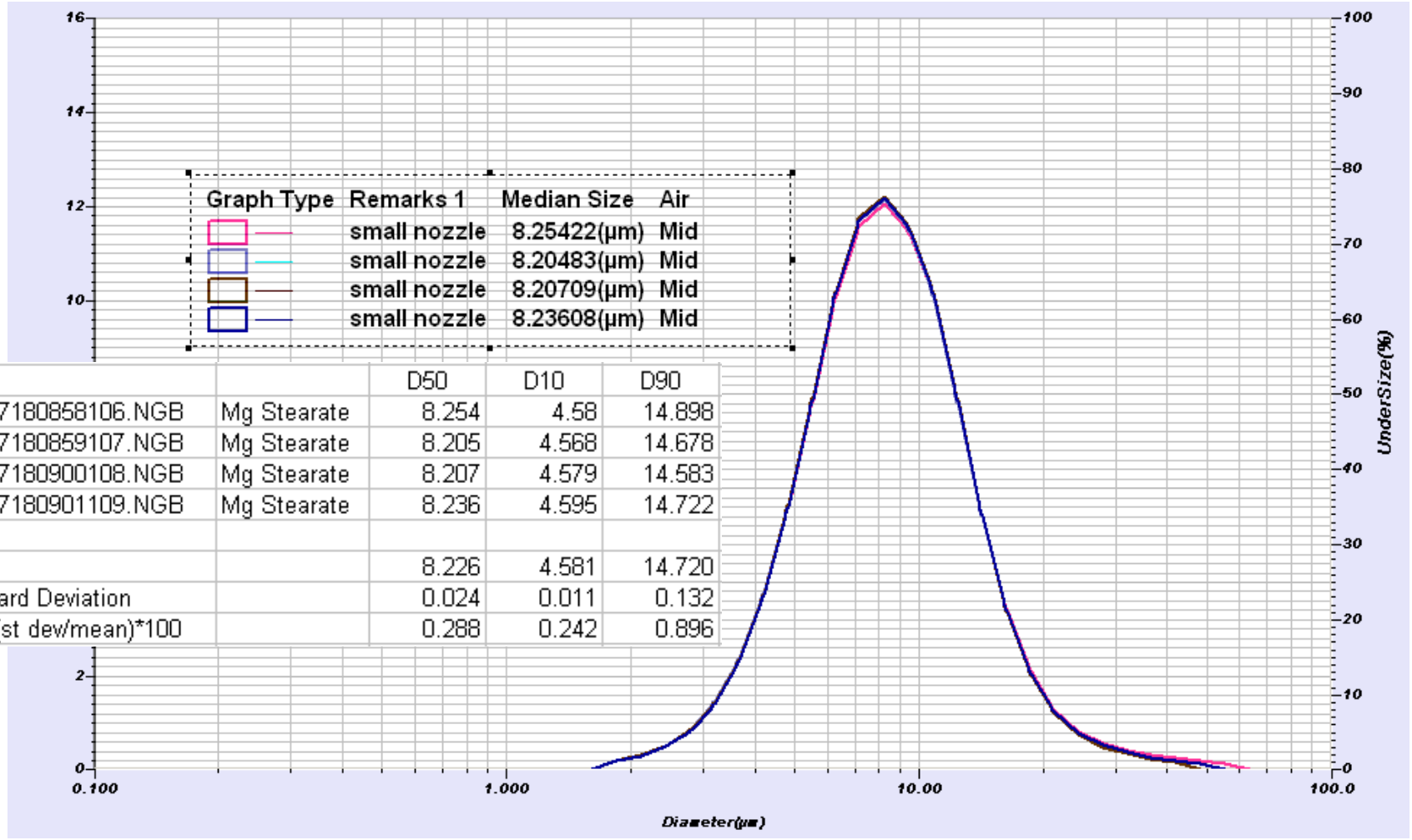
Repeatability: Target T%

Use feedback for adjusting feeder speed to ensure consistent sample feed.

More repeatable results.
Less operator dependence.



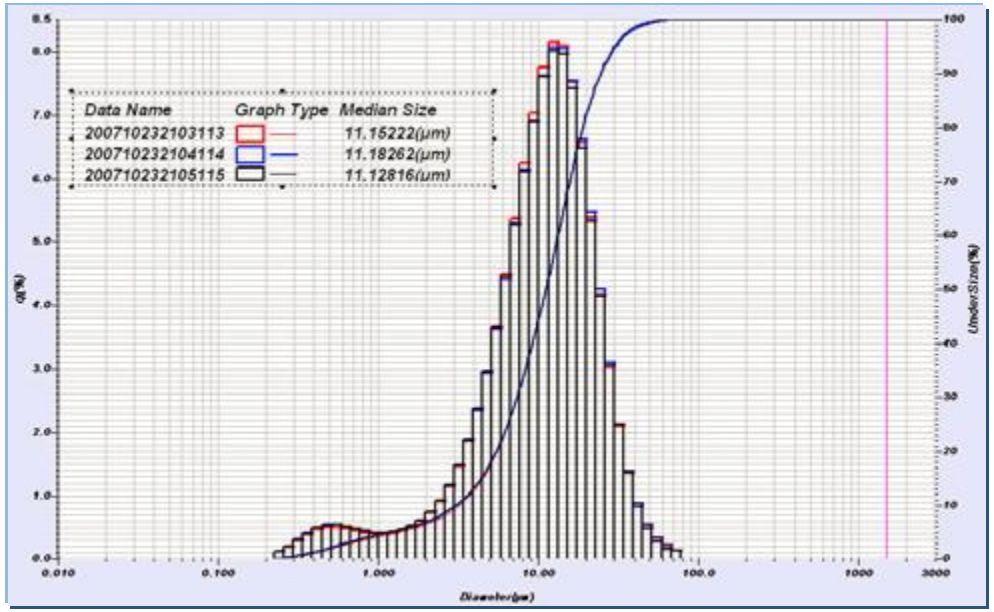
Reproducibility– Mg Stearate dry, 2 bar



		D50	D10	D90
200707180858106.NGB	Mg Stearate	8.254	4.58	14.898
200707180859107.NGB	Mg Stearate	8.205	4.568	14.678
200707180900108.NGB	Mg Stearate	8.207	4.579	14.583
200707180901109.NGB	Mg Stearate	8.236	4.595	14.722
Mean		8.226	4.581	14.720
Standard Deviation		0.024	0.011	0.132
COV (st dev/mean)*100		0.288	0.242	0.896

Reproducibility: Dry cement

	D10	D50	d90
Portland Cement 1	3.255	11.152	24.586
Portland Cement 2	3.116	11.183	24.671
Portland Cement 3	3.112	11.128	24.92
Average	3.161	11.154	24.726
Std. Dev.	0.082	0.027	0.173
CV (%)	2.6	0.24	0.70

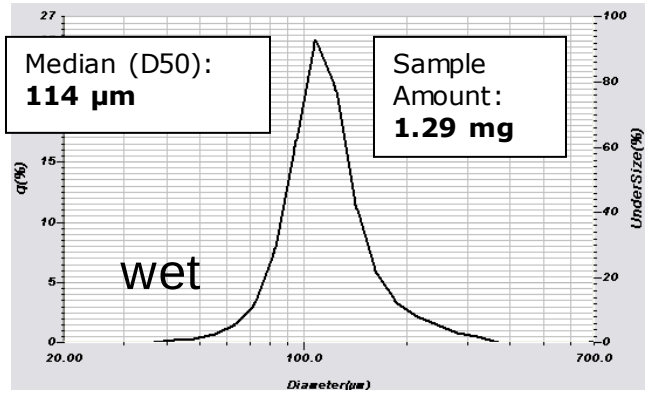


How much sample?

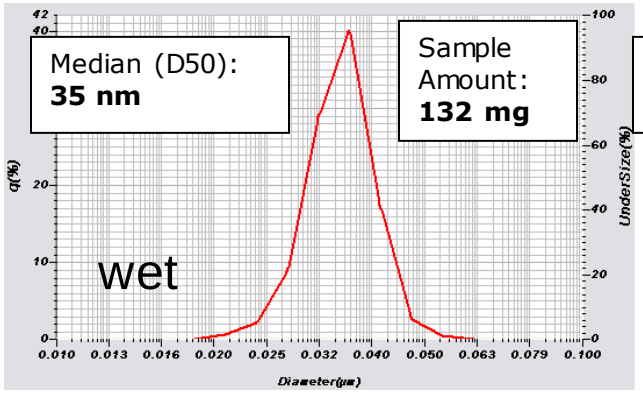
It depends on sample …..

Larger, broad distributions require larger sample quantity

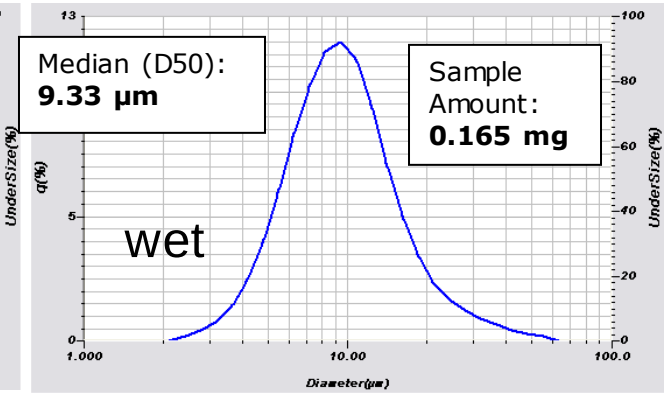
Dry can measure less than 5 mg (over a number of particle sizes).



Bio polymer



Colloidal silica



Magnesium stearate

Concluding comments

There are a lot of options for sample handling and the right choice can make measurements better.

Option	Comments	Volume (mL)
LiterFlow	Wide distributions, rarely used	1000
Solvo/AquaFlow	Most common, most extensions	180-250
MiniFlow	Still has on board dispersion	35-50
Fraction cell	Precious samples, ~10 micron maximum size	5-15
HL / paste cells	High viscosity and high concentration samples	0.5-2
Dry powder feeder	No liquids, ~1 micron maximum size due to surface energy	n/a