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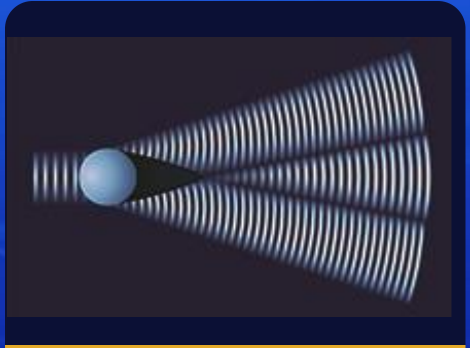
Explore the future

HORIBA Scientific

Modern Particle Characterization Techniques Series

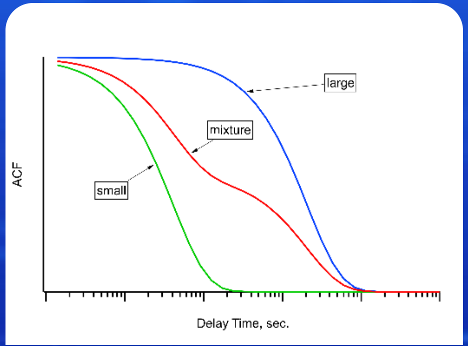
Dr. Mike Pohl

I: Introduction



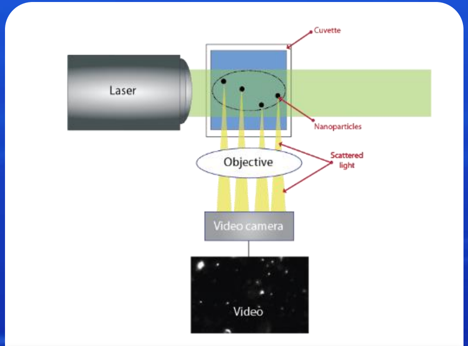
Julie Chen Nguyen

II: Laser Diffraction



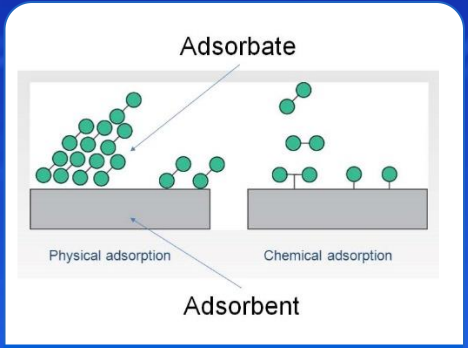
Dr. Jeff Bodycomb

III: DLS



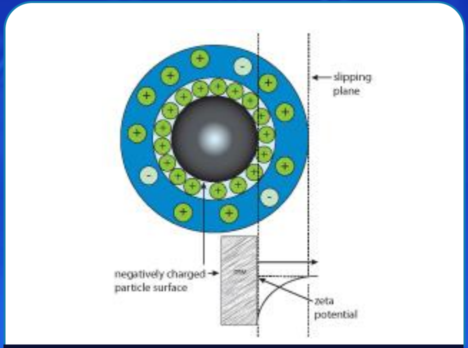
Dr. Sean Travers

IV: Multi-laser NTA



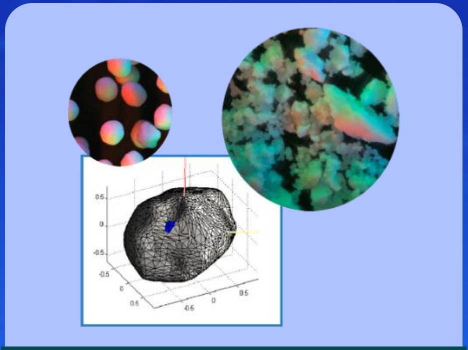
Carl Lundstedt

Series V: BET



Dr. David Fairhurst

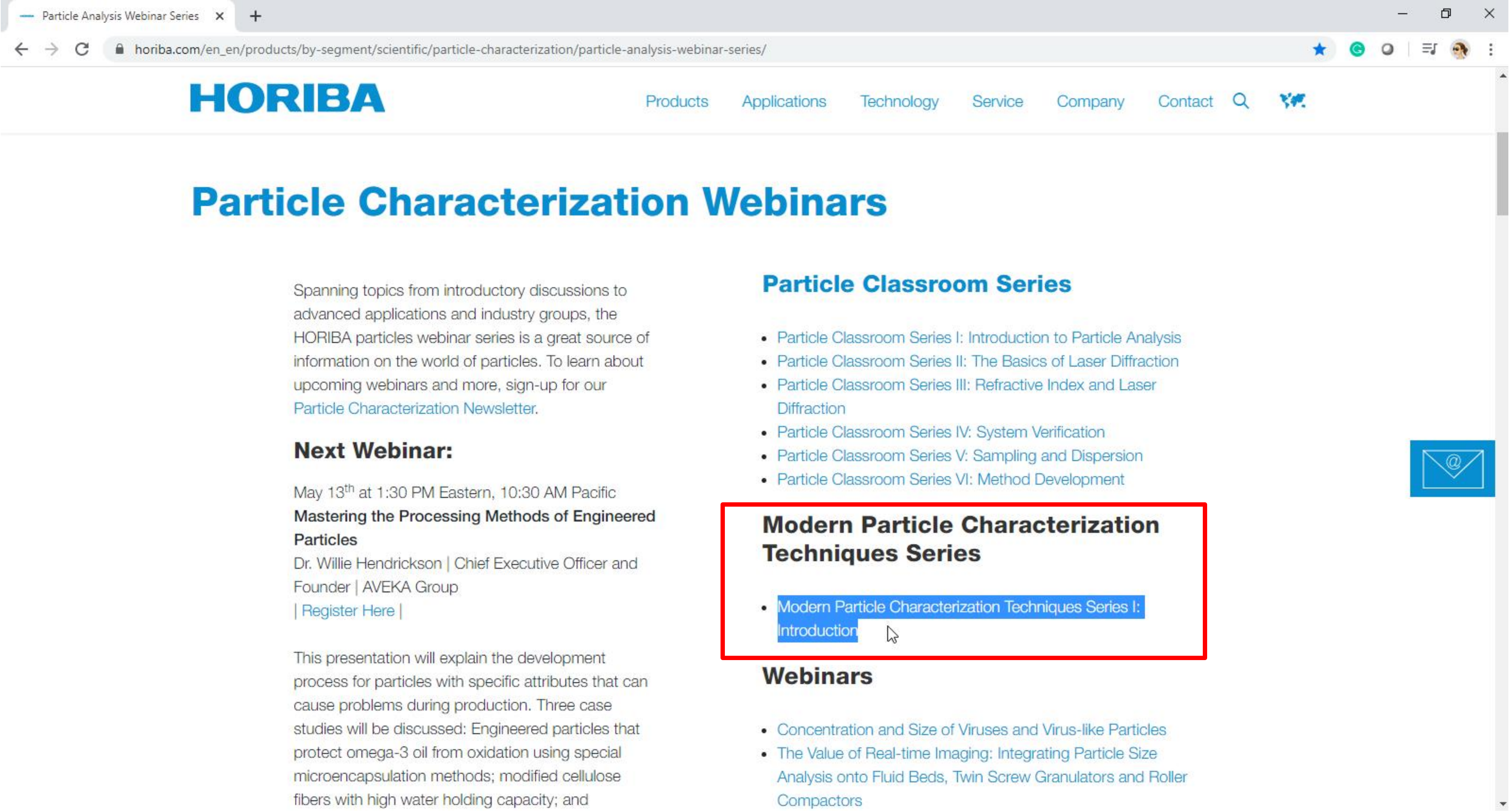
VI: Zeta Potential



Darren McHugh

VII: Image Analysis

Look for the introduction webinar here…



Particle Analysis Webinar Series

horiba.com/en_en/products/by-segment/scientific/particle-characterization/particle-analysis-webinar-series/

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Products Applications Technology Service Company Contact

Particle Characterization Webinars

Spanning topics from introductory discussions to advanced applications and industry groups, the HORIBA particles webinar series is a great source of information on the world of particles. To learn about upcoming webinars and more, sign-up for our Particle Characterization Newsletter.

Next Webinar:

May 13th at 1:30 PM Eastern, 10:30 AM Pacific
Mastering the Processing Methods of Engineered Particles
Dr. Willie Hendrickson | Chief Executive Officer and Founder | AVEKA Group
| Register Here |

This presentation will explain the development process for particles with specific attributes that can cause problems during production. Three case studies will be discussed: Engineered particles that protect omega-3 oil from oxidation using special microencapsulation methods; modified cellulose fibers with high water holding capacity; and

Particle Classroom Series

- Particle Classroom Series I: Introduction to Particle Analysis
- Particle Classroom Series II: The Basics of Laser Diffraction
- Particle Classroom Series III: Refractive Index and Laser Diffraction
- Particle Classroom Series IV: System Verification
- Particle Classroom Series V: Sampling and Dispersion
- Particle Classroom Series VI: Method Development

Modern Particle Characterization Techniques Series

- Modern Particle Characterization Techniques Series I: Introduction

Webinars

- Concentration and Size of Viruses and Virus-like Particles
- The Value of Real-time Imaging: Integrating Particle Size Analysis onto Fluid Beds, Twin Screw Granulators and Roller Compactors

HORIBA Instruments Incorporated

Irvine, California

Julie Chen Nguyen

Modern Particle Characterization Techniques Series II

Laser Diffraction

May 28, 2020

Overview

- **Intro to laser diffraction (LD) technique**
  - Mie vs. Fraunhofer
- **Instrument translation**
  - What makes LD a “Modern” technique?
  - LA-960, LA-350 and the accessories
- **Sampling**
- **Wet dispersion**
  - Demo video
  - Case studies
  - Method development
- **Dry dispersion**
  - Demo video
  - Case studies
  - Method development
- **Selecting an appropriate optical model**
  - Method Expert
- **Conclusion**
- **Q&A**

NIST definition of a particle

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“Any condensed-phase tridimensional discontinuity in a dispersed system may generally be considered a particle; e.g., droplets in an emulsion or solids dispersed in a liquid….An aggregate may also be regarded as a particle”

Importance of particles?

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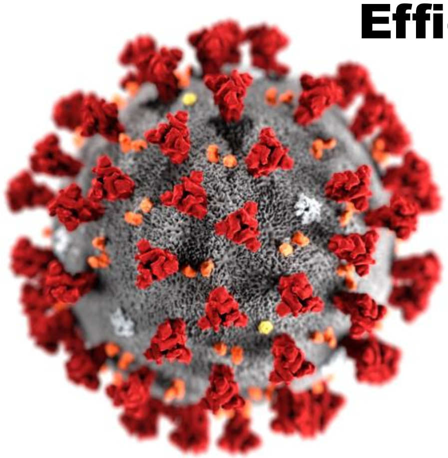
Efficacy

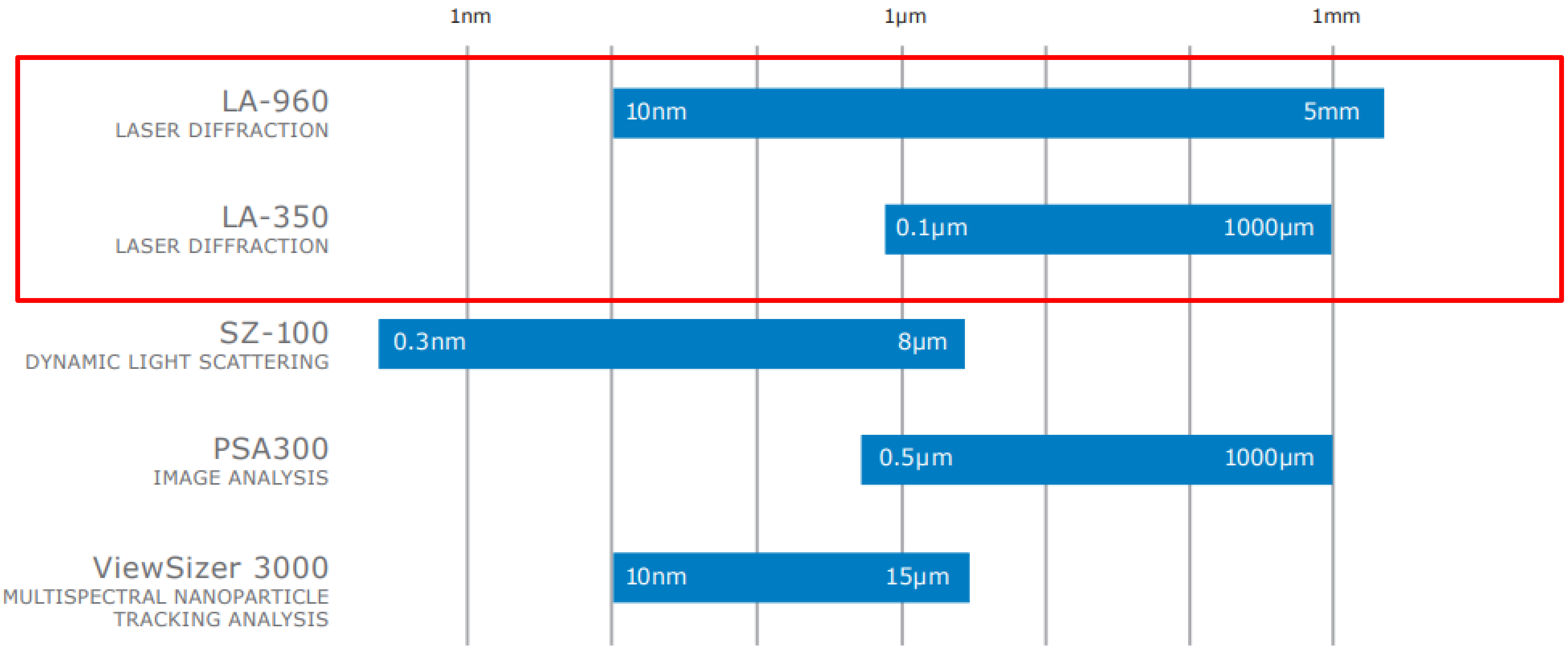


Stability

www.horiba.com/partice > Download Center > Applications Note

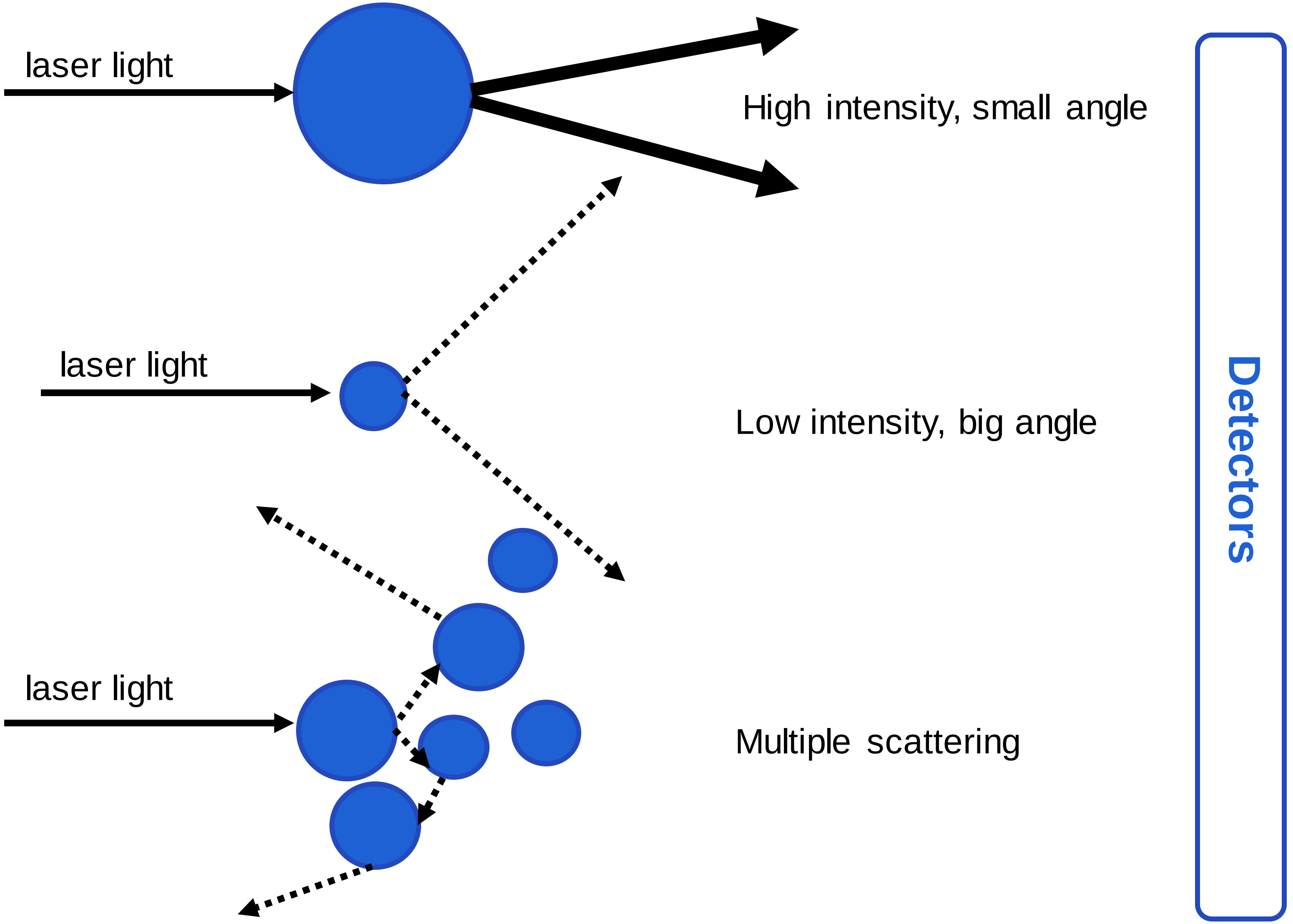
Power

Perspective



Diffraction Pattern

1. Intensity
2. Angle



When a light beam strikes a particle...

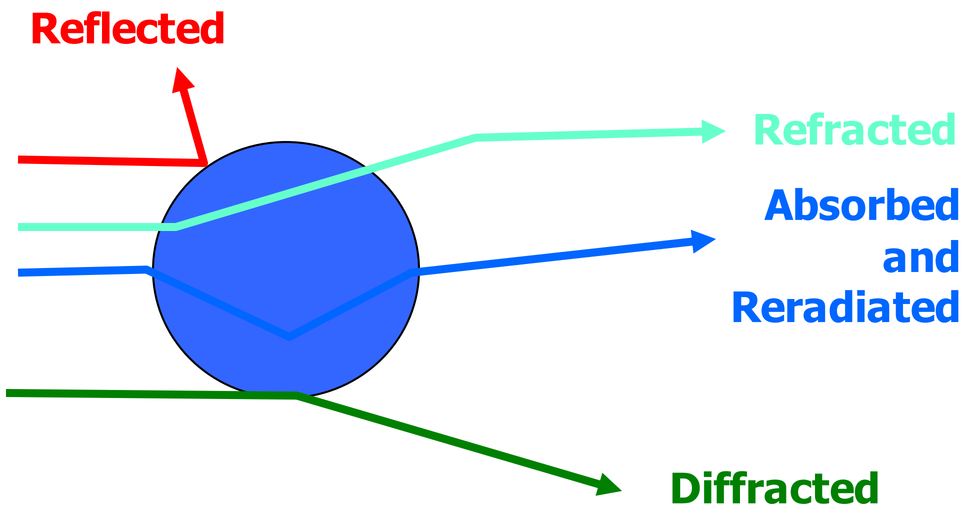
Some of the light is:

Diffracted

Reflected

Refracted

Absorbed and Reradiated



- Small particles require knowledge of optical properties:
  - Real Refractive Index (bending of light, wavelength of light in particle)
  - Imaginary Refractive Index (absorption of light within particle)
  - Refractive index values less significant for large particles

Optical Models – your choice

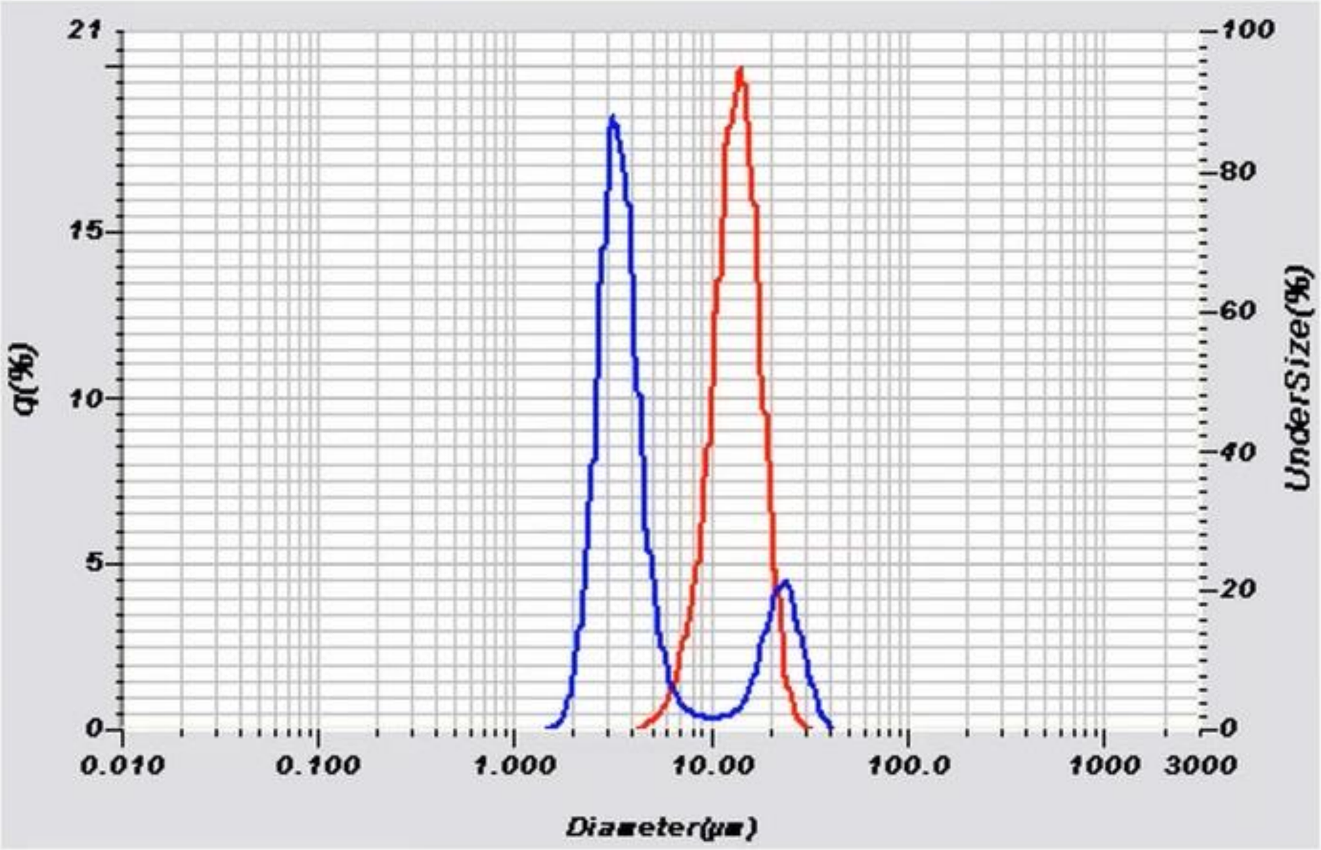
- Fraunhofer approximation
  - Opaque (absorbs light completely)
  - 2-D circular disks
  - Particles > 50 microns (ISO 13320 Section A.5)
  - Particle is much larger than the wavelength of light (only diffraction is considered)

- Mie Theory
  - 3-D spheres and optically homogeneous
  - Refractive index of particle and surrounding medium is known

Webinar: Interpreting Laser Diffraction Results for Non-Spherical Particles by Dr. David M. Scott

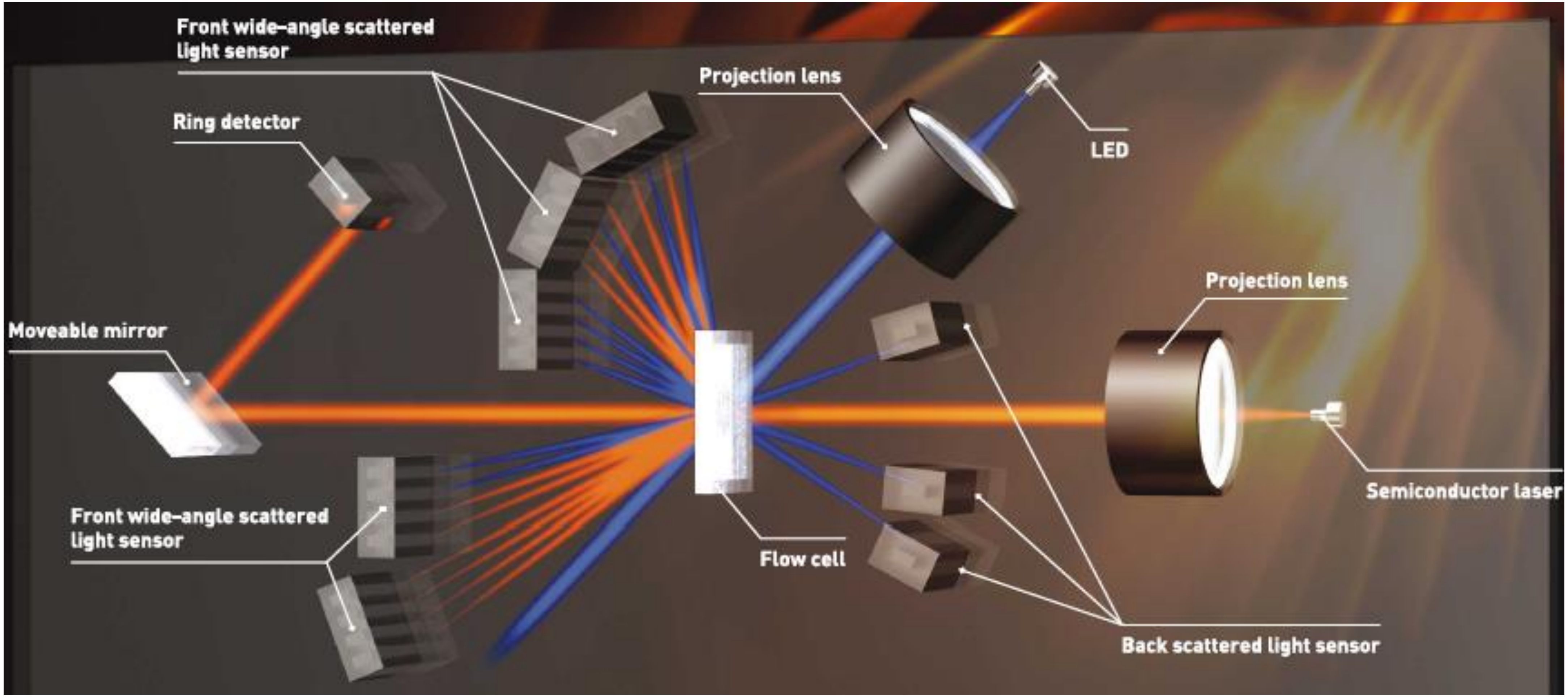
Mie vs. Fraunhofer: Glass beads

Data Name	Graph Type	Refractive Index (R)
Standard Glass Beads Mie	red	STD-GLASSBEADS[STD-GLASSBEADS(1.510 - 0.000i),
Standard Glass Beads Fraunhofer	blue	Fraunhofer Kernel[Fraunhofer Kernel(0.000 - 0.000i

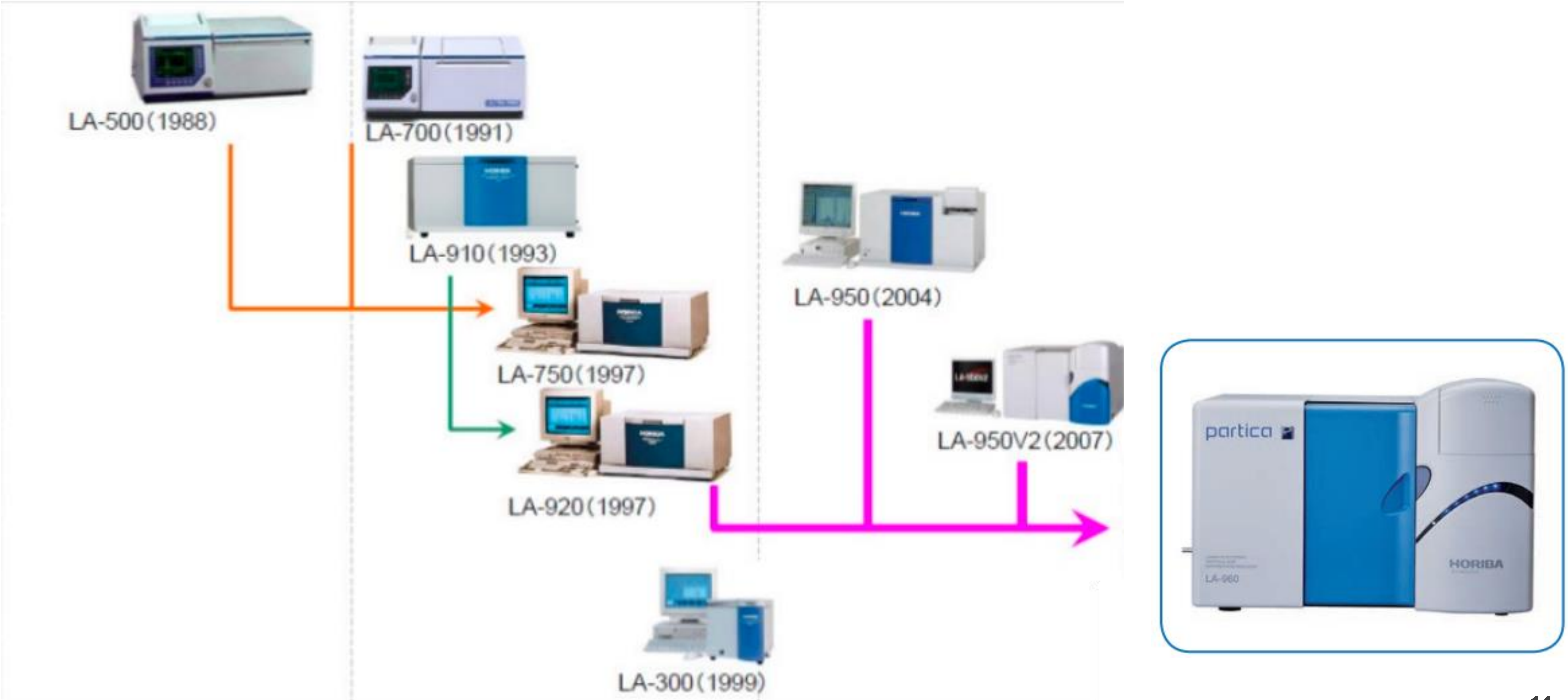


Graph Type	D(v,0.1)	D(v,0.5)	D(v,0.9)
red	8.98783(µm)	13.47741(µm)	18.8536
blue	2.58072(µm)	3.62044(µm)	22.3174

The optics (LA-960)



What makes LD a “Modern” technique?



Instrument translation

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	LA-960	LA-350
Technology	Laser Diffraction	Laser Diffraction
Measurement Output	Particle Size	Particle Size
Measurement Range	0.01 µm to 5000 µm	0.1 µm to 1000 µm
Typical Sample Amount*	10 mg to 5 g	10 mg to 5 g
External Dimensions	705 x 565 x 500 mm	297 x 429 x 376 mm
Light Source/ Resolution	605 nm Laser Diode 405 nm LED	605 nm Laser Diode

Accessories for wet analysis

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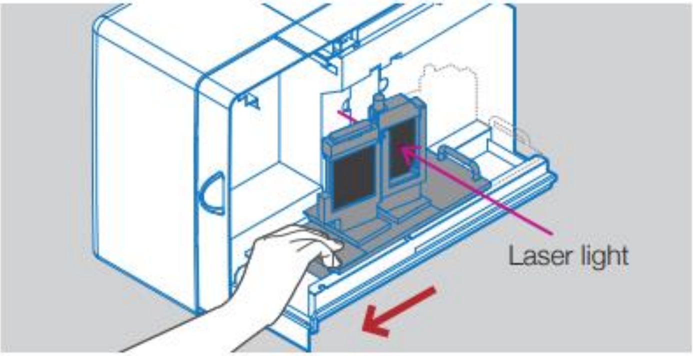
■ Easy cell switching.



Change cells just sliding changer table. No tools required.

Flow Cell

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

Mini flow

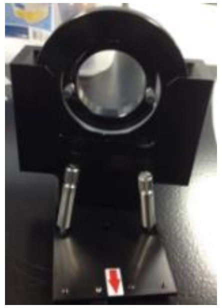
Volume (mL)	35
Ranger (µm)	0.01 – 1,000

Fraction Cell

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

Paste Cell

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



HL Cell

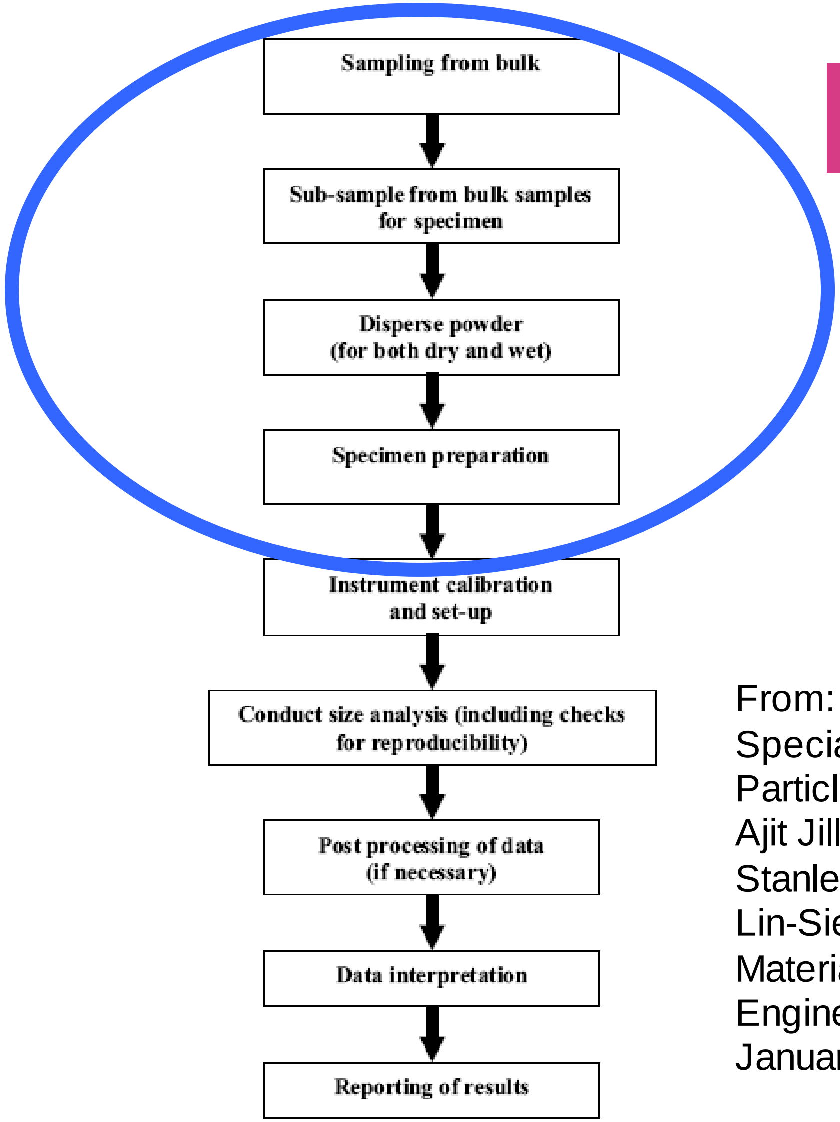
Volume (mL)	0.5 – 2 mL
Ranger (µm)	0.01 – 500

Accessories for dry analysis

Dry Unit

Pressure (MPa)	**0 – 0.4**
Ranger (µm)	**0.1 – 5,000**

Particle analysis workflow



Particle Classroom Series V: Sampling and Dispersion by Dr. Jeff Bodycomb

From: NIST Recommended Practice Guide
Special Publication 960-1
Particle Size Characterization
Ajit Jillavenkatesa
Stanley J. Dapkunas
Lin-Sien H. Lum
Materials Science and
Engineering Laboratory
January 2001

Reliability of selected sampling methods using a 60:40 sand mixture

Sampling Technique	Standard Deviation
Cone and Quartering	6.81
Scoop Sampling	5.14
Table Sampling	2.09
Chute Slitting	1.01
Spinning Riffling	0.146
Random Variation	0.075

Allen, T. (1997). Particle Size Measurement Volume 1: Powder Sampling and Particle Size measurement fifth edition., Page 21. Chapman & Hall.

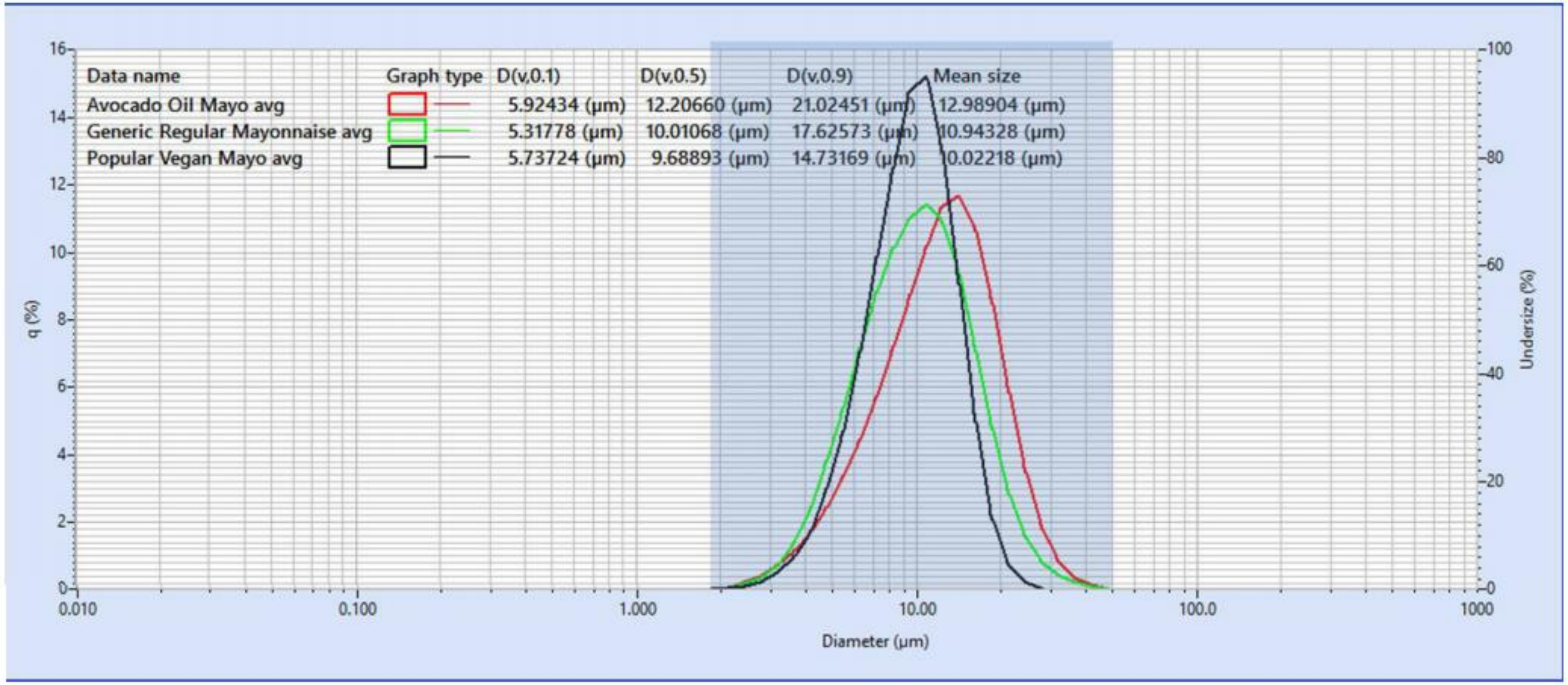
Wet analysis demo video

Case Study: Mayonnaise Oil in Water (O/W) Emulsion

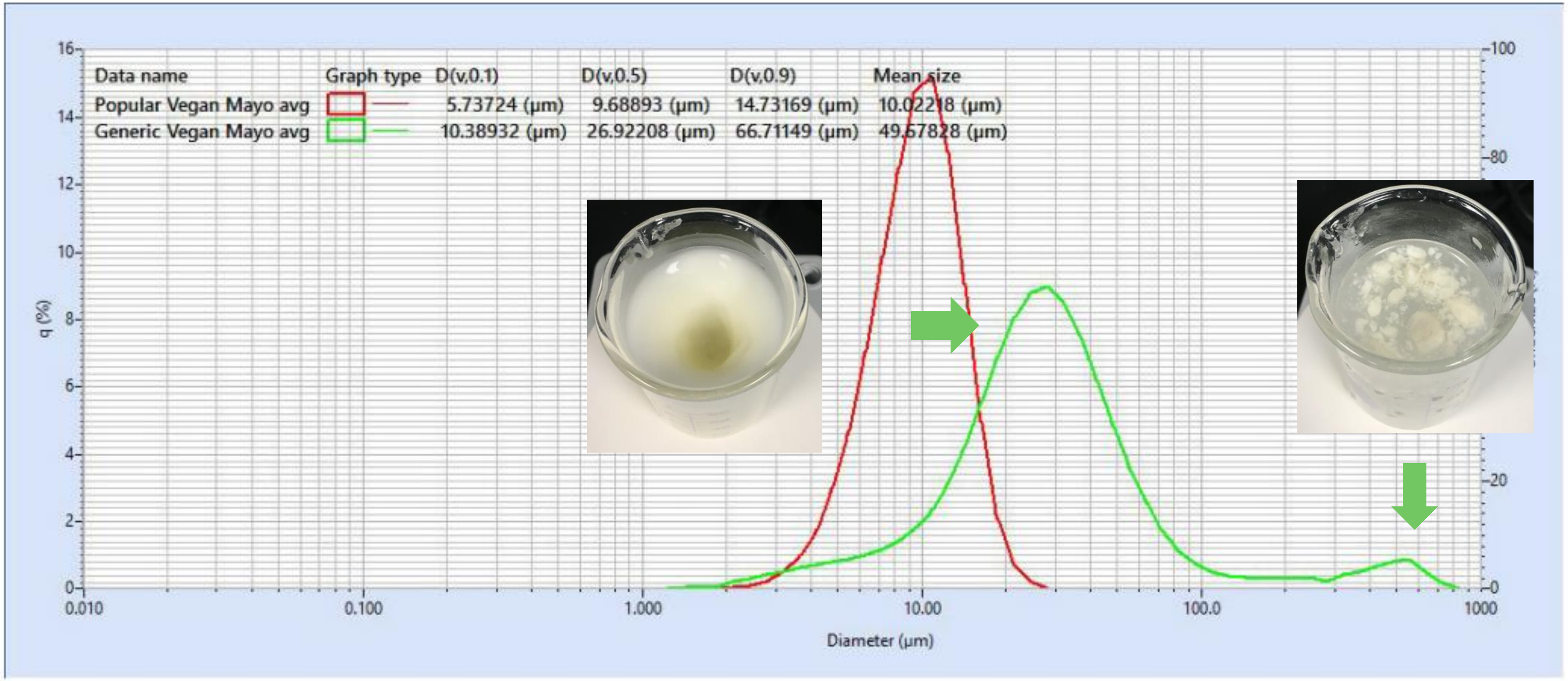
- Oil (dispersed phase) + vinegar (continuous phase) + egg yolk (emulsifier) + salt (taste)
- Avoid canola oil and stick with “healthy fat” trend such as extra virgin olive oil, avocado, and almonds.*
- Physiochemical properties
  - Emulsion stability
  - Rheological properties
  - Sensory characteristics
  - **Particle size distribution**
  - pH
  - Cholesterol content
  - microstructures

References:
*Hyman, Mark. *Eat Fat, Get Thin*. New York: Little Brown and Company, 2016. Print pg.77 *Key TJ, Appleby PN, Davey GK, Allen NE, Spencer EA, Travis RC. Mortality in British Vegetarians: review and preliminary result from EPIC-Oxford. Am J Clin Nutr 2003 Sep;

Case Study: Mayonnaise Oil in Water (O/W) Emulsion

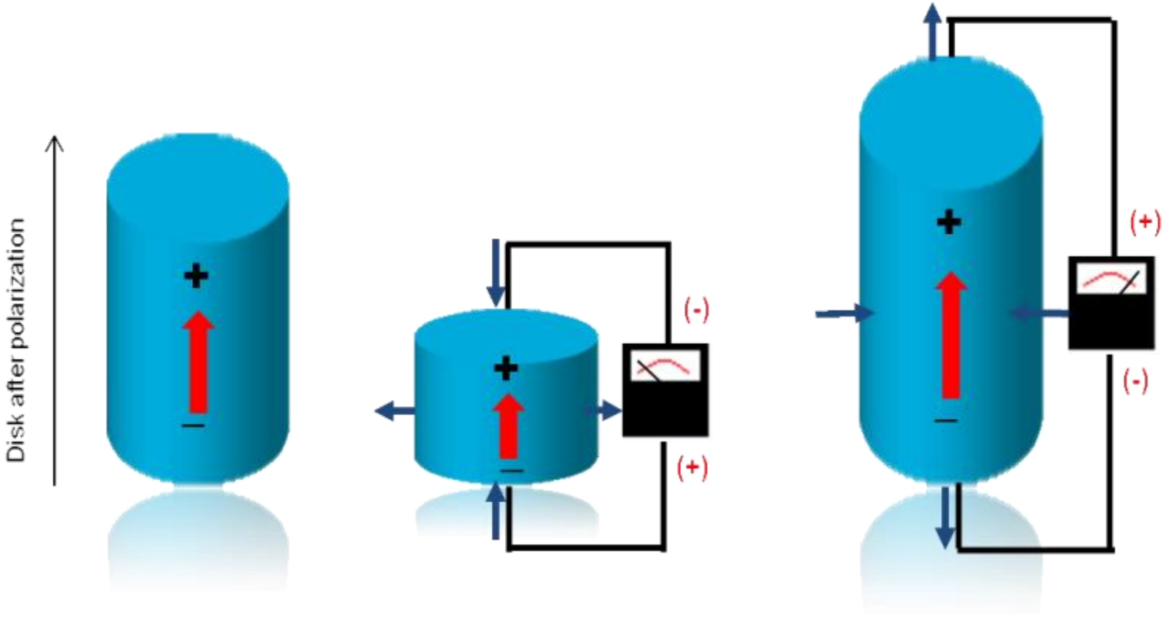


Case Study: Mayonnaise Oil in Water (O/W) Emulsion



Case Study: Pieoelectric Particles

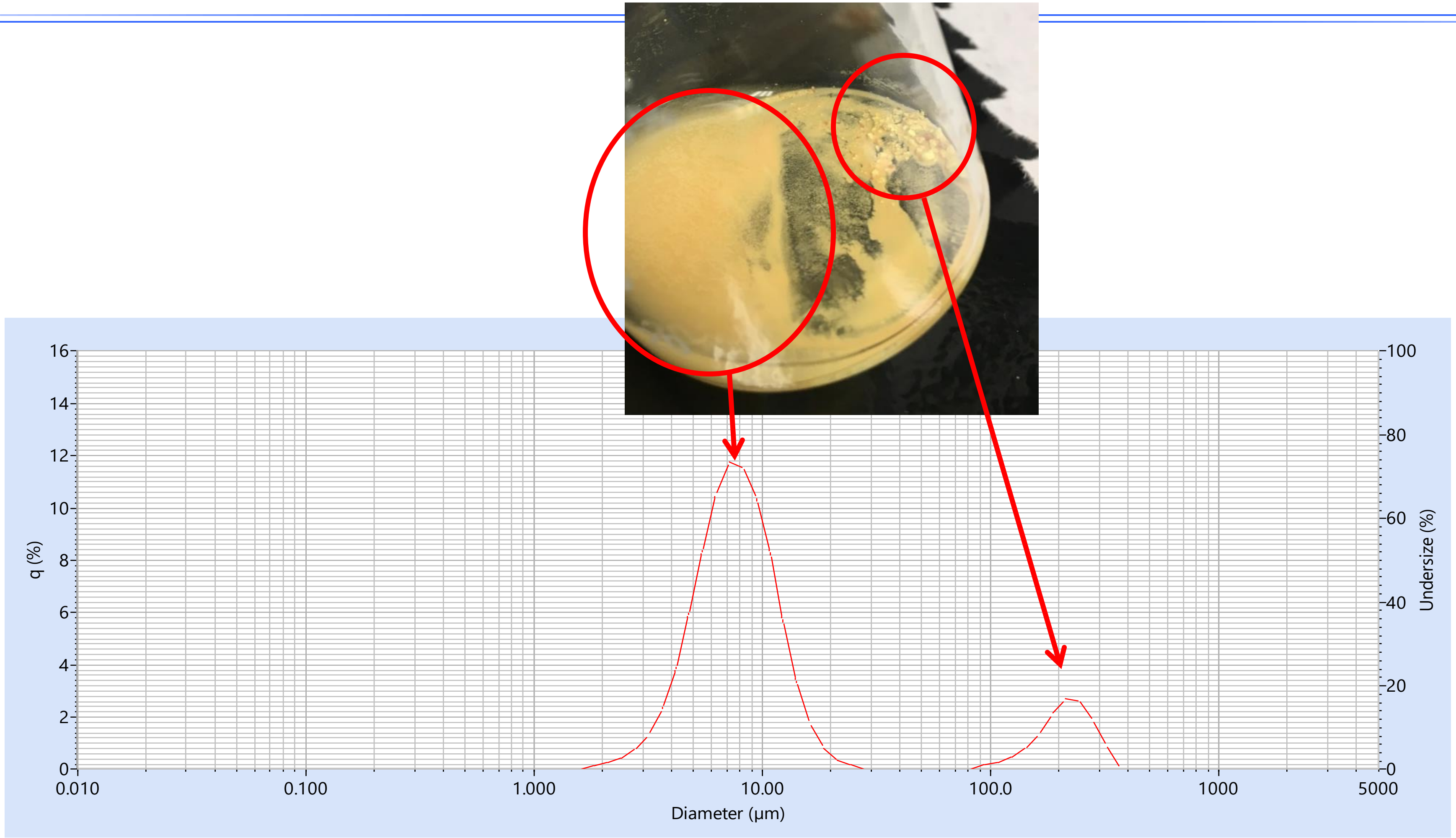
- Piezoelectricity was discovered in 1880
- Mechanical stress can generate electrical charges and vice versa
  - Ultrasound
  - DPI



Cyclic expansion and contraction of piezoelectric material

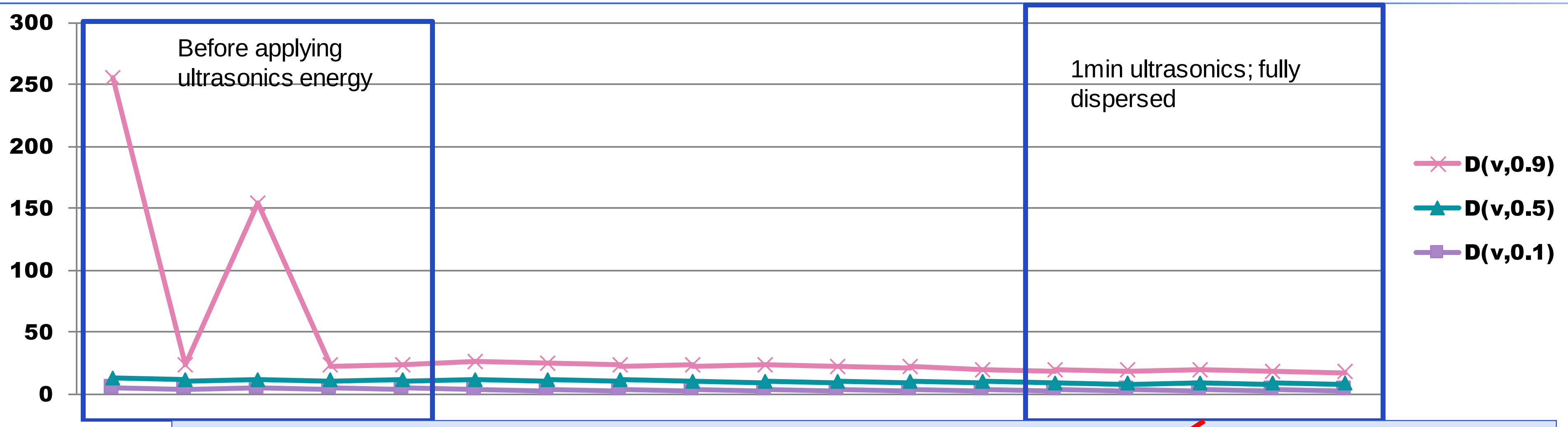
- Dispersion method: water with surfactant (e.g. 0.1wt% of Darven C or 0.1wt% of Sodium Pyrophosphate solution)
- Refractive Index 2.20-1i
- RI is important for particles <50um (ISO13320)

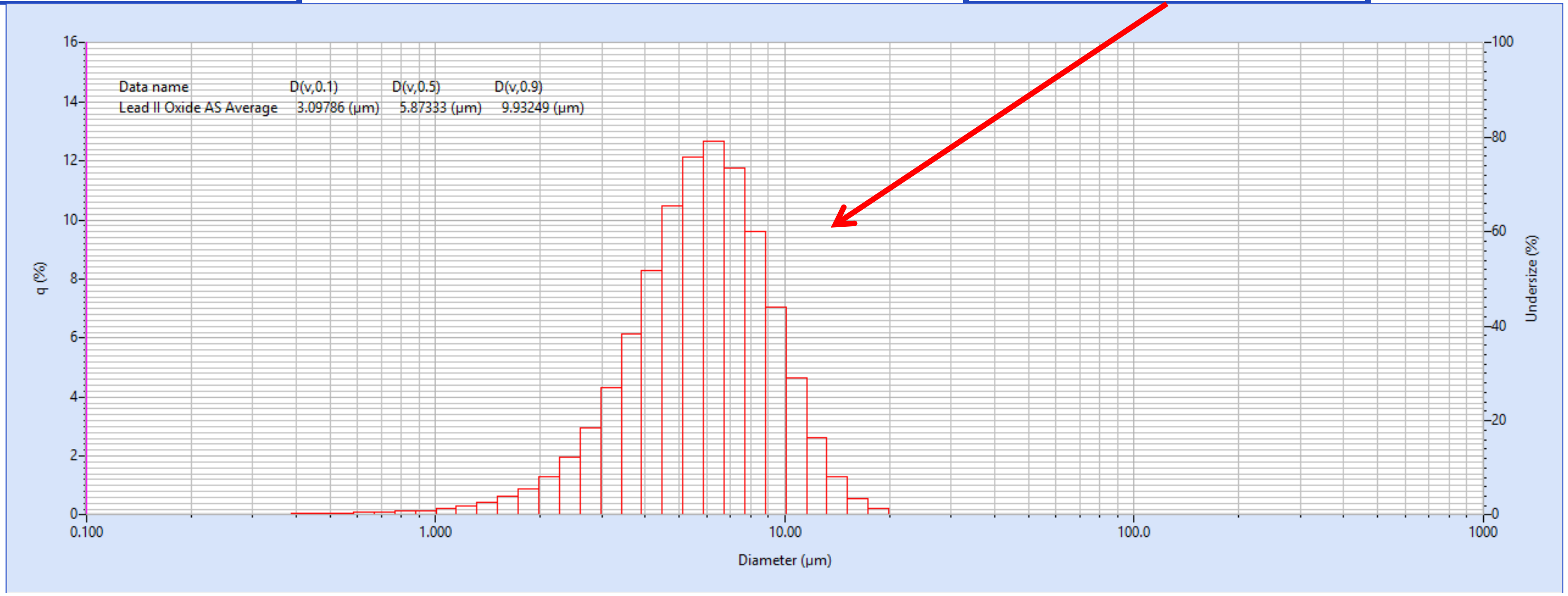
Case Study: Lead (II) Oxide



Special thanks to Harris Corp for providing samples and insights of the PZT manufacturing process, www.exelispzt.com

Method Development: Wet





Method Development: Wet

- Sampling (>100um)
- Wetting (surface tension)
- Ultrasonic energy?
- Sample concentration?
  - Typically 95-80 transmission % is good
- Measurement time?
- Pump/stirring speed?

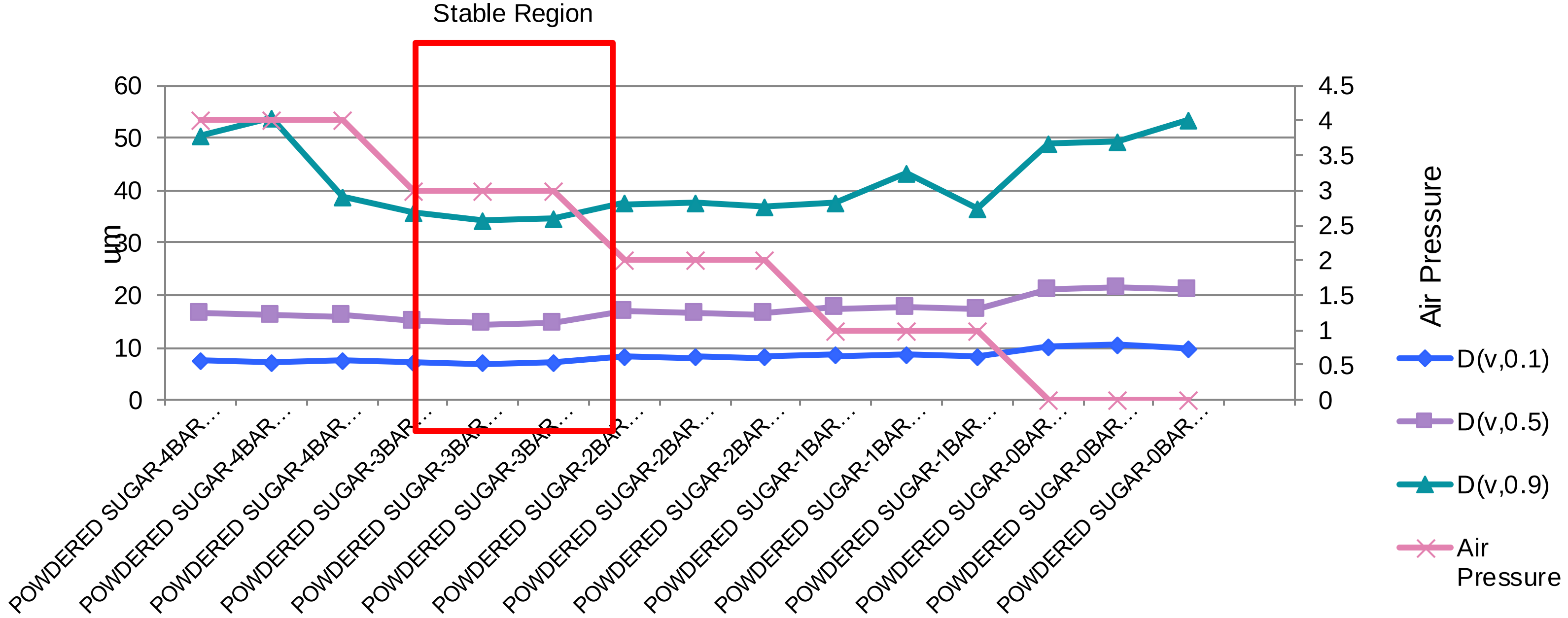
Dry analysis demo video

Case Study: Powdered Sugar

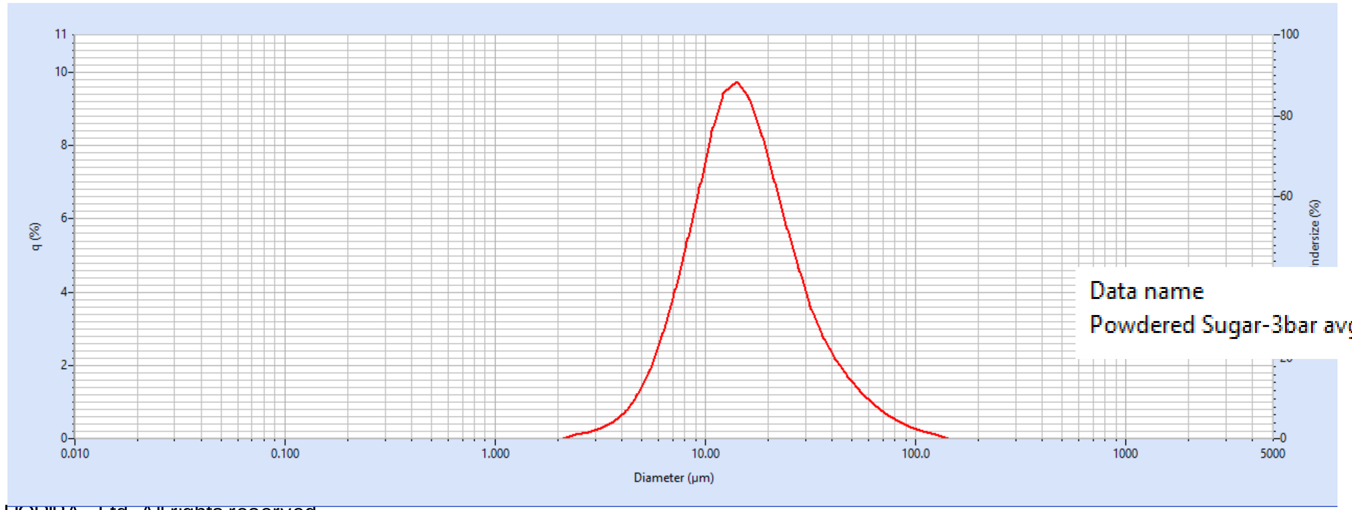
- (Hammer) Milled* from white granulated crystals (Application notes AN141, AN175)
  - 2X – defined as 82%<200mesh (74um)
  - 4X- defined as 92%<200mesh
  - 6X – defined as 93.5% <200mesh
  - 8X – defined as 96% <200mesh
  - **10X – defined as 98%<200mesh**
  - 12X – defined as 98% <325mesh (45um)
  - Silk Sugar – defined as 97% <20.5um
- Production goals:
  - The efficiency of milling – from pilot size to full production
  - Narrow particle size distributions
    - Uniformity minimizes separation
    - Dissolution/mixing
  - **Flowability** (anti-caking agent 3%)
- Dry Dispersion:
  - Sampling (>100um)
  - Energy – (Pressure Size Titration test)
  - Slope Dv90>Dv50>Dv10

Source: *http://www.hmicronpowder.com/industries/food/sugar

Method Development: Dry



Diameter (um)	ASTM Mesh	Frequency %	Cum %
20	635	69.071	69.071
25	500	10.644	79.715
32	450	8.164	87.879
38	400	3.865	91.744
45	325	2.768	94.512
53	270	1.872	96.383
63	230	1.417	97.801
75	200	0.983	98.783
90	170	0.649	99.432
106	140	0.331	99.763
125	120	0.186	99.949
150	100	0.051	100

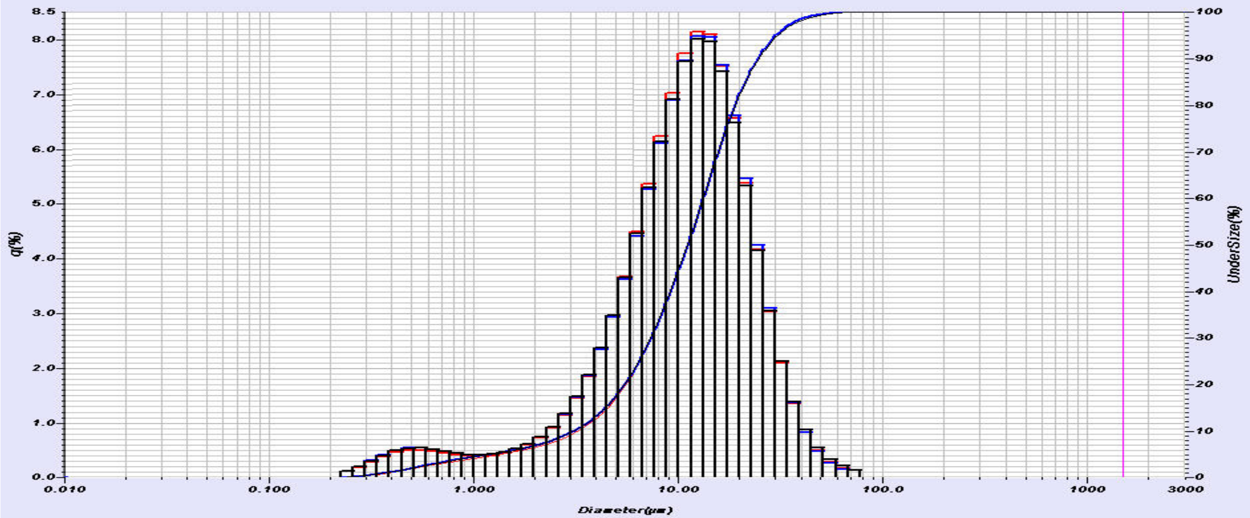


Data name	Graph type	D(v,0.1)	D(v,0.5)	D(v,0.9)	Cumulative % at diameter(8)
Powdered Sugar-3bar avg		7.31500 (μm)	14.89861 (μm)	34.84077 (μm)	(8)74.00 (μm)- 98.718(%)

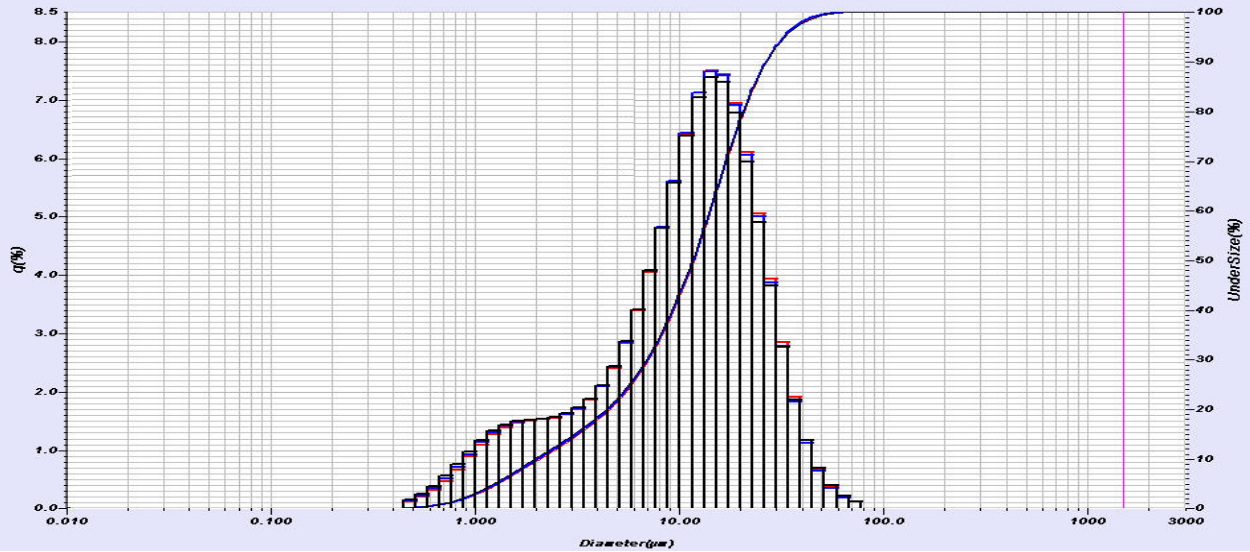
Method Development: Dry

- Sampling (>100um)
- Air pressure?
  - Pressure Size Titration test
- Sample concentration?
- Measurement time?
- Tray vibration speed?

Wet and dry dispersion agreement: Cement



File Name	Dv10	Dv50	Dv90
LA-960 Dry Cement_1	3.256	11.152	24.586
LA-960 Dry Cement_2	3.116	11.183	24.671
LA-960 Dry Cement_3	3.112	11.128	24.92
Average	3.161	11.154	24.726
Std. Dev.	0.082	0.027	0.173
CV(%)	2.589	0.245	0.701
ISO13320-1	5	3	5

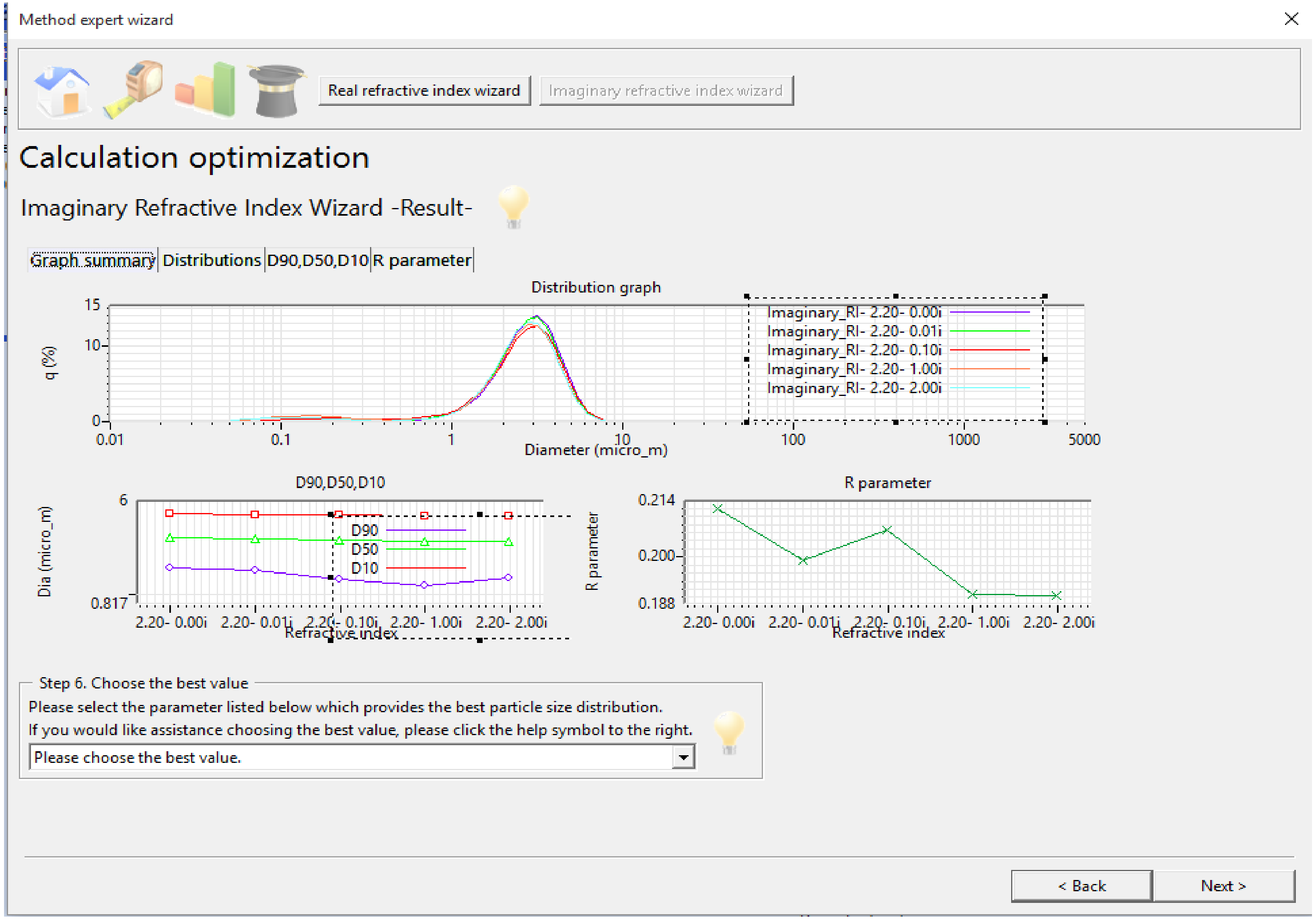


File Name	Dv10	Dv50	Dv90
LA-960 Wet Cement_1	2.122	11.81	27.047
LA-960 Wet Cement_2	2.058	11.696	26.743
LA-960 Wet Cement_3	1.999	11.614	27.001
Average	2.06	11.707	26.93
Std. Dev.	0.062	0.098	0.164
CV(%)	2.996	0.838	0.607
ISO13320-1	5	3	5

Selecting Appropriate Optical Properties

- Real component of the RI:
  - Look it up (Google)
  - Measure it (refractometer)
  - 2 decimal places
- Imaginary component of the RI:
  - Established imperially or by experience
  - 0i is reserved for standards and emulsions
  - Practically, >0i are required to accommodate for surface roughness
  - 0i, 0.001i, 0.01i, 0.1i, 1i

RI Assessment - Lead Zirconate



Concluding Comments

- Repeatability
  - ISO13320
  - Built-in software function
- Reproducibility
  - USP <420>
  - EP 2.9.31
- Robustness

More Concluding Comments

- Wide size range
  - Most advanced analyzer measures from 10 *nano* to 5 *milli*
- Flexible sample handlers
  - Powders, suspensions, emulsions, pastes, creams
- Very fast
  - Allows for high throughput, 100’s of samples/day
- Easy to use
  - Many instruments are highly automated with self-guided software
- Good design = Excellent precision
  - Reduces unnecessary investigation/downtime
- First principle measurement
  - No calibration necessary
- Massive global install base/history

What to know more about this particle series?
Sign up for the newsletter:

labinfo@horiba.com

Modern Particle Characterization Techniques Series III:

Thank you

Omoshiro-okashiku
Joy and Fun

Terima kasih
நன்றி
Gracias
谢谢
Tack ska du ha
Danke
Σας ευχαριστώ πάρα πολύ
धन्यवाद
Grazie
THANK YOU
شُكْرًا
ขอบคุณครับ
Obrigado
Большое спасибо
ありがとうございました
Cảm ơn
Merci
감사합니다
Dziękuję