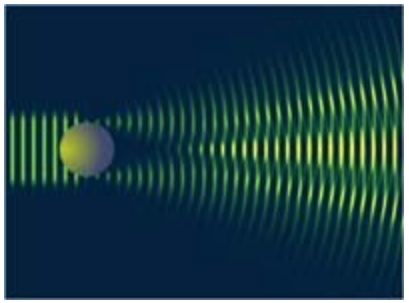
Introduction to Laser Diffraction

Fundamental Principles and Everyday Uses



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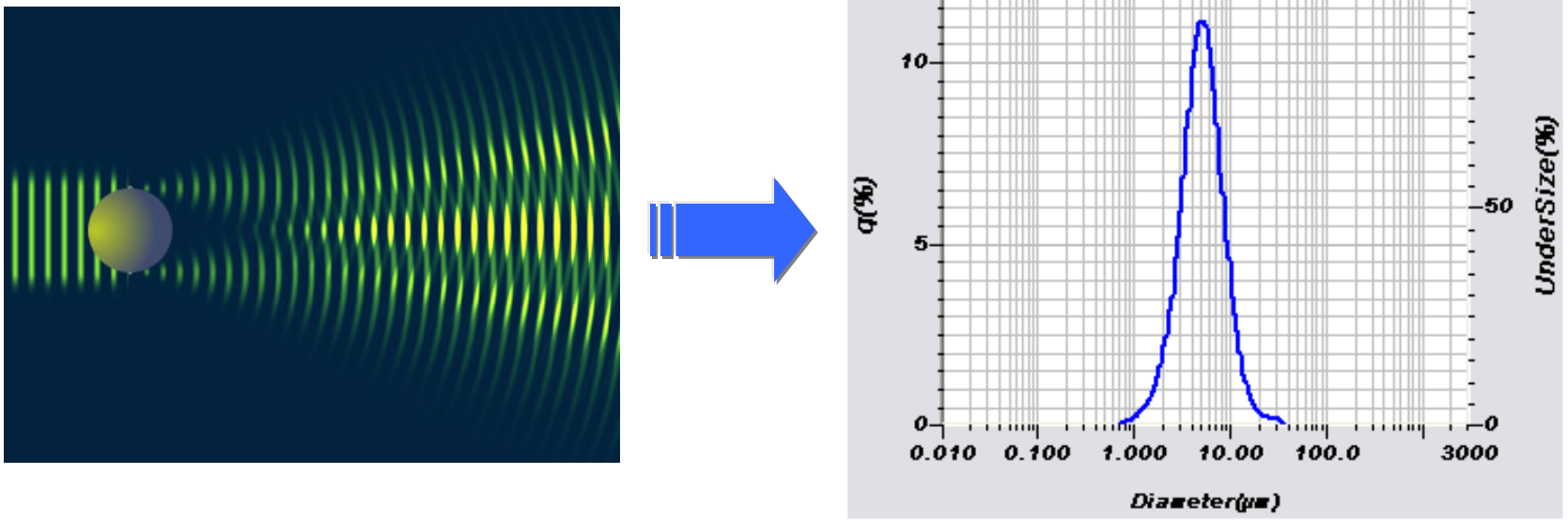
Plan of Attack

- How does it work?
- What can diffraction do?
- How does it help you?
- Strong points, weak spots
- Q&A

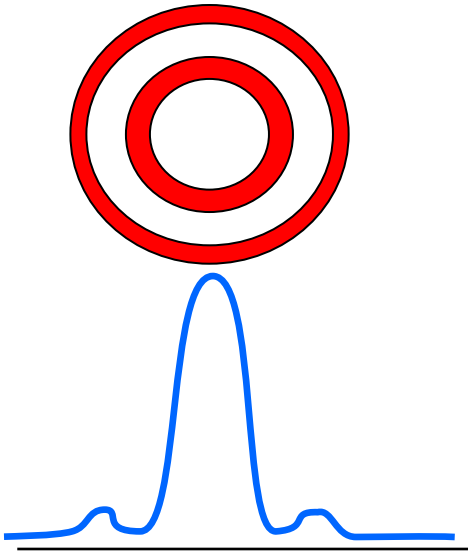
Core Principle

- Can investigate a particle with light and derive its size

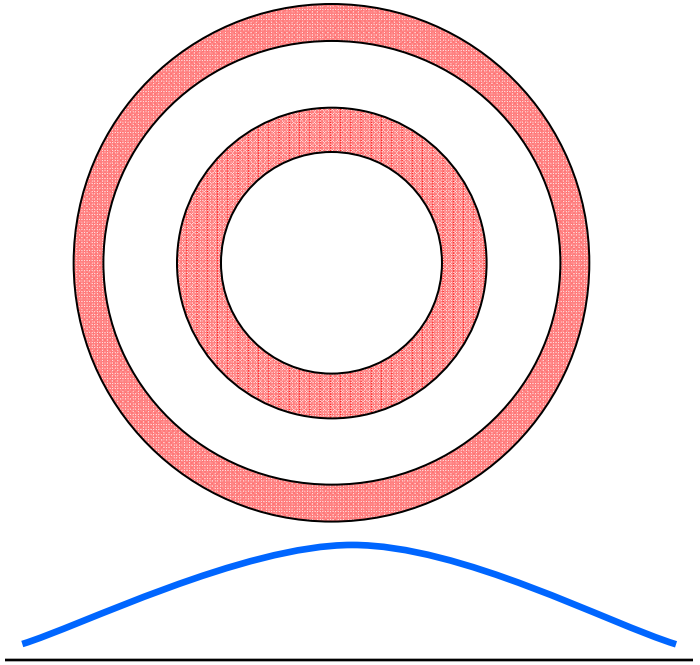


Core Principle

- Why? Because the angle and intensity of the scattered light depends on its size



Larger Particle



Smaller Particle

Core Principle

- So all we need is a light source, a particle, light detectors, and a German mathematician

Joseph von Fraunhofer

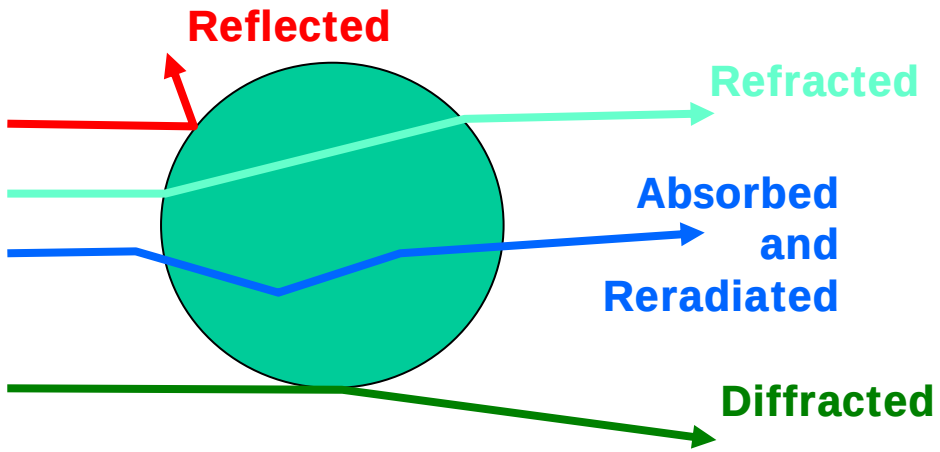
or

Gustav Mie

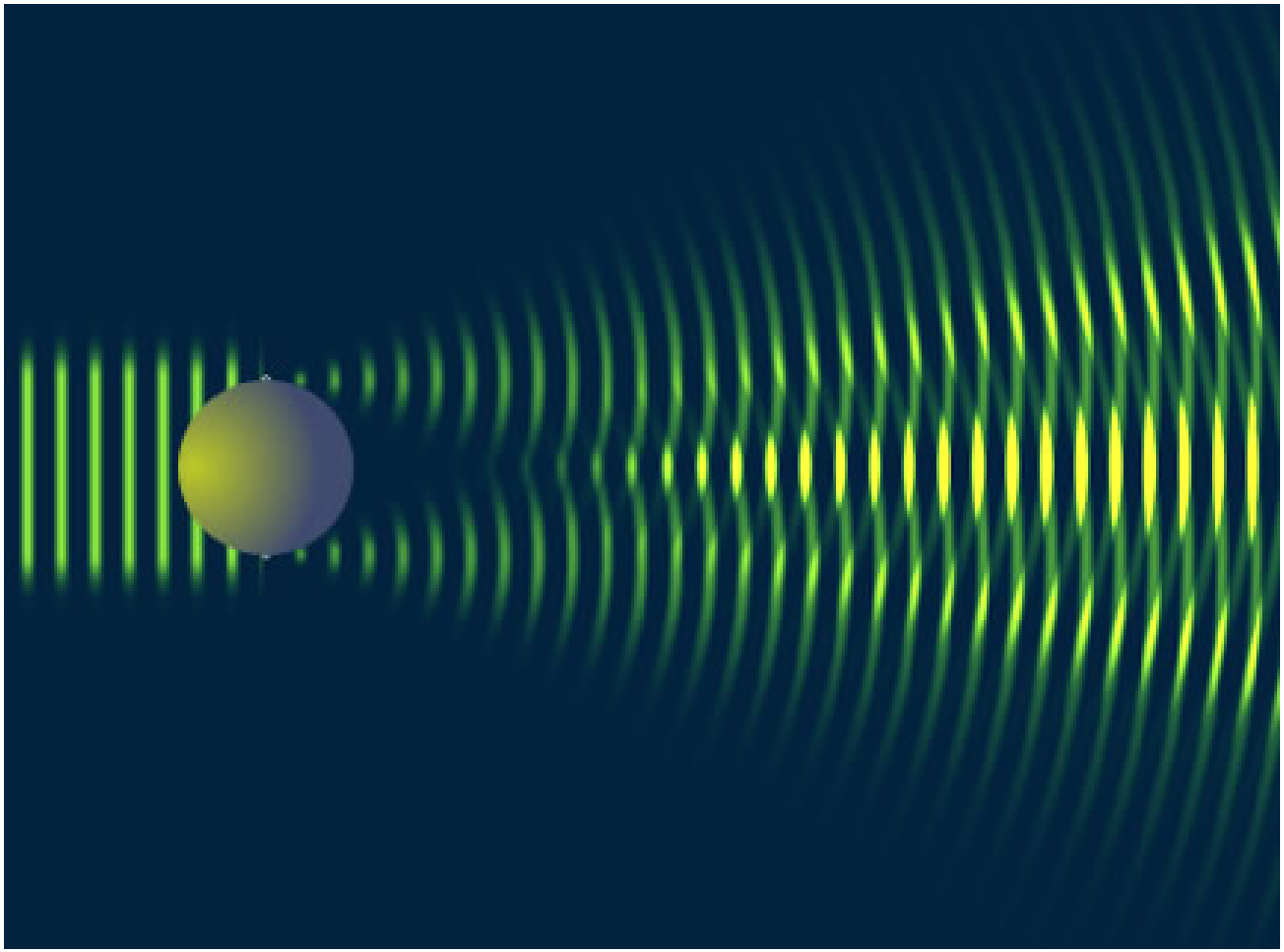
…but this guy has a better solution

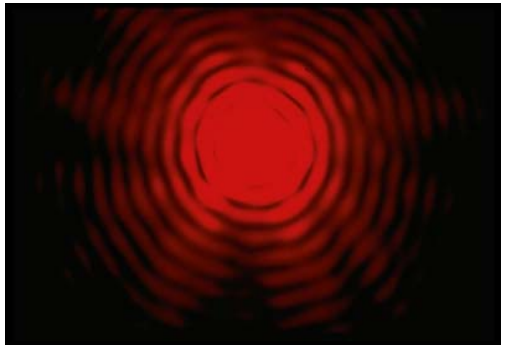
Four Types of Interaction

- Diffraction
- Refraction
- Reflection
- Absorption

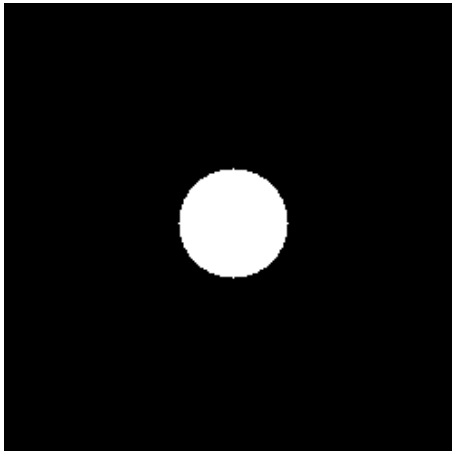
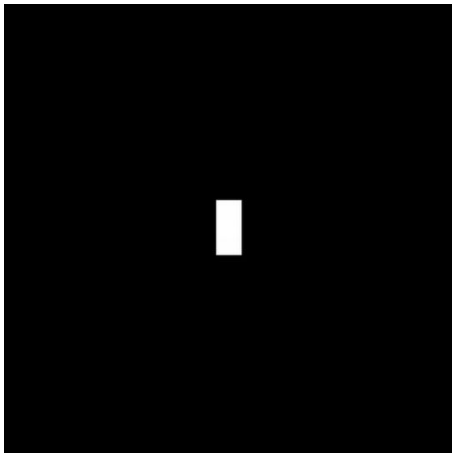


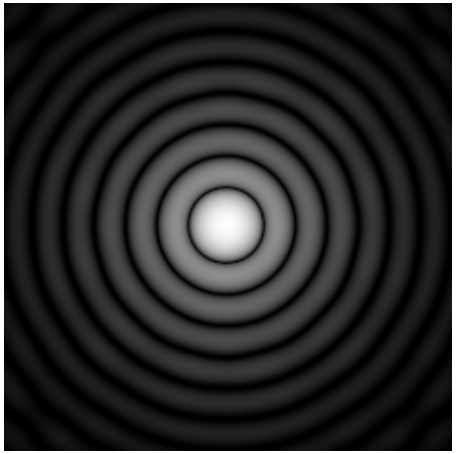
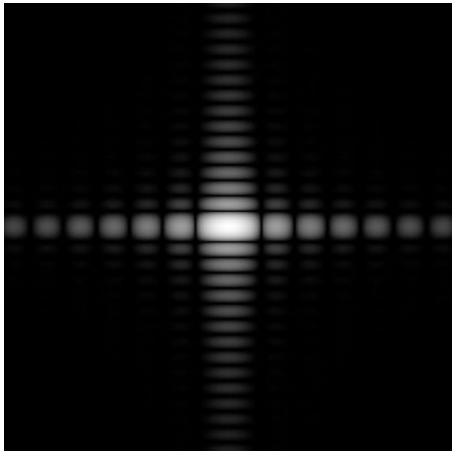
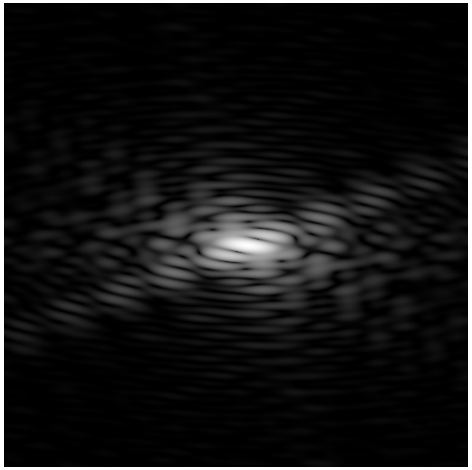
Edge Diffraction





Diffraction Patterns

Why a “pattern”?

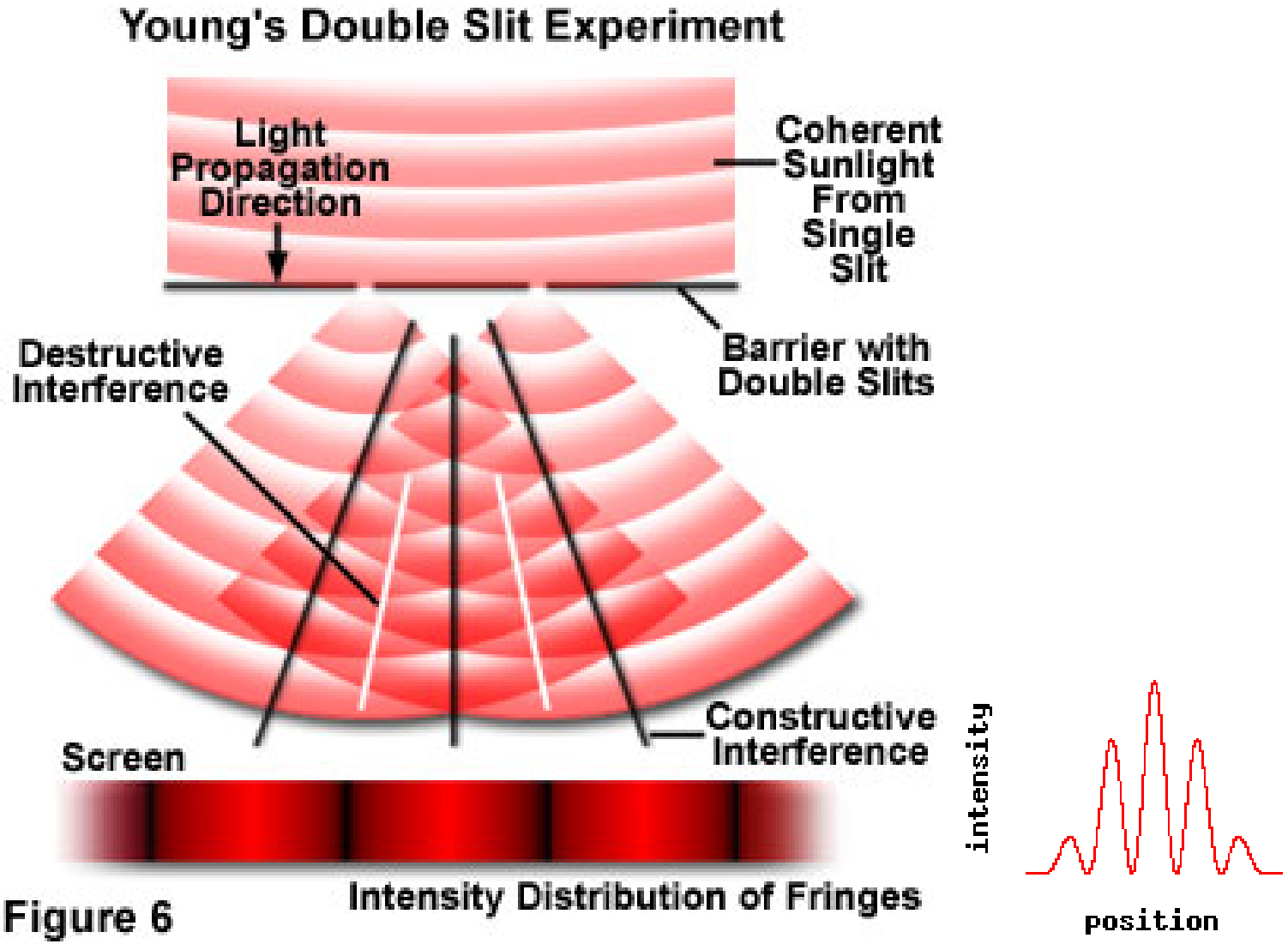
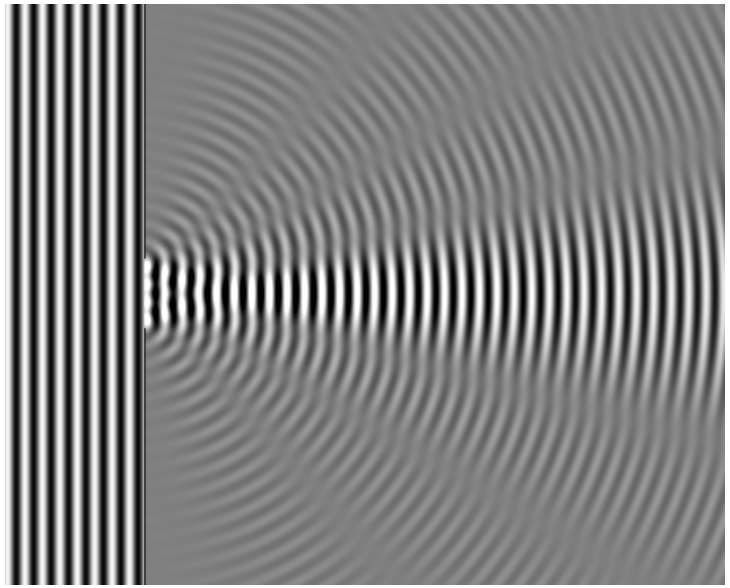
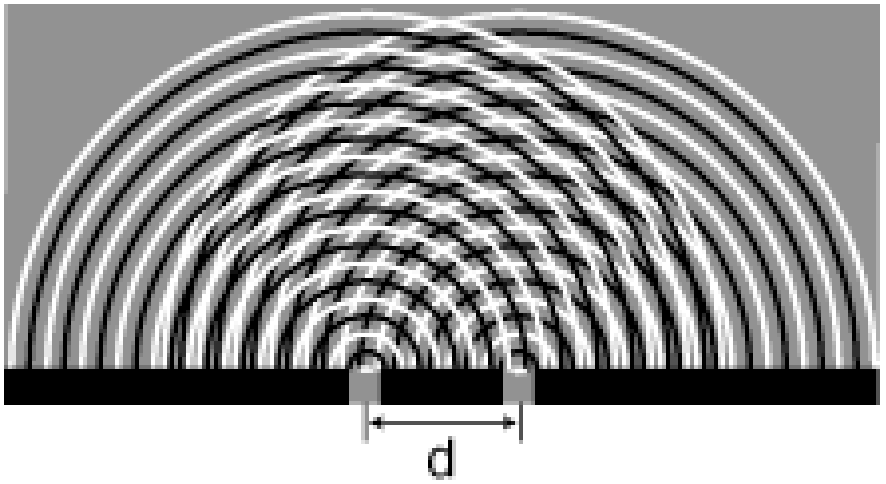


Figure 6

Double Slit = Single Slit

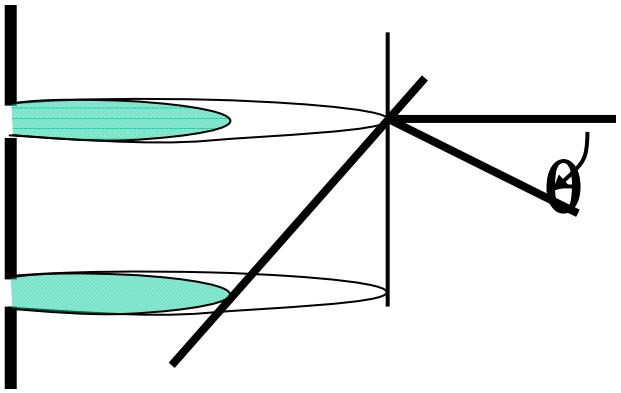


Single slit diffraction pattern

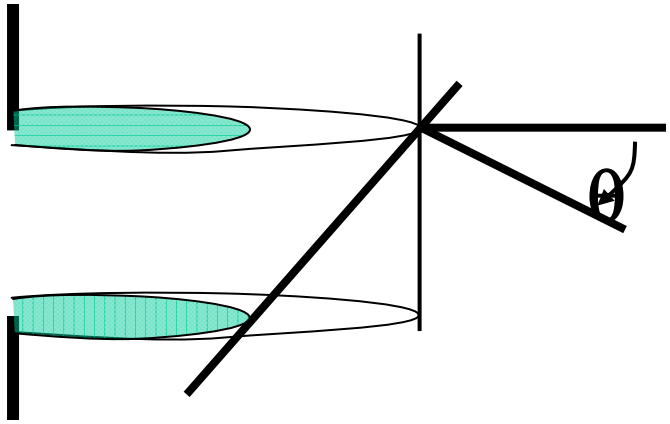


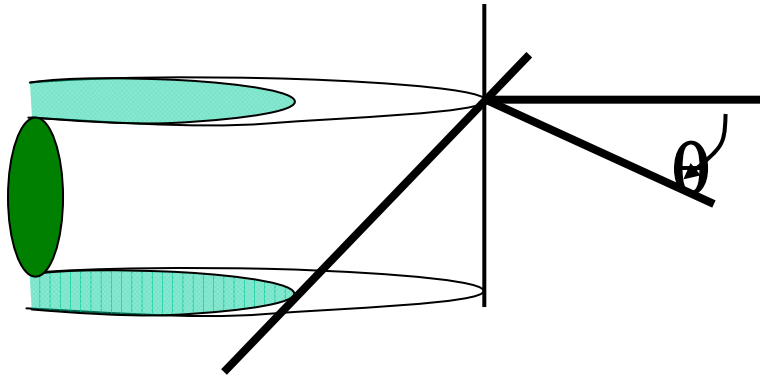
Double slit diffraction pattern

A “particle” in disguise



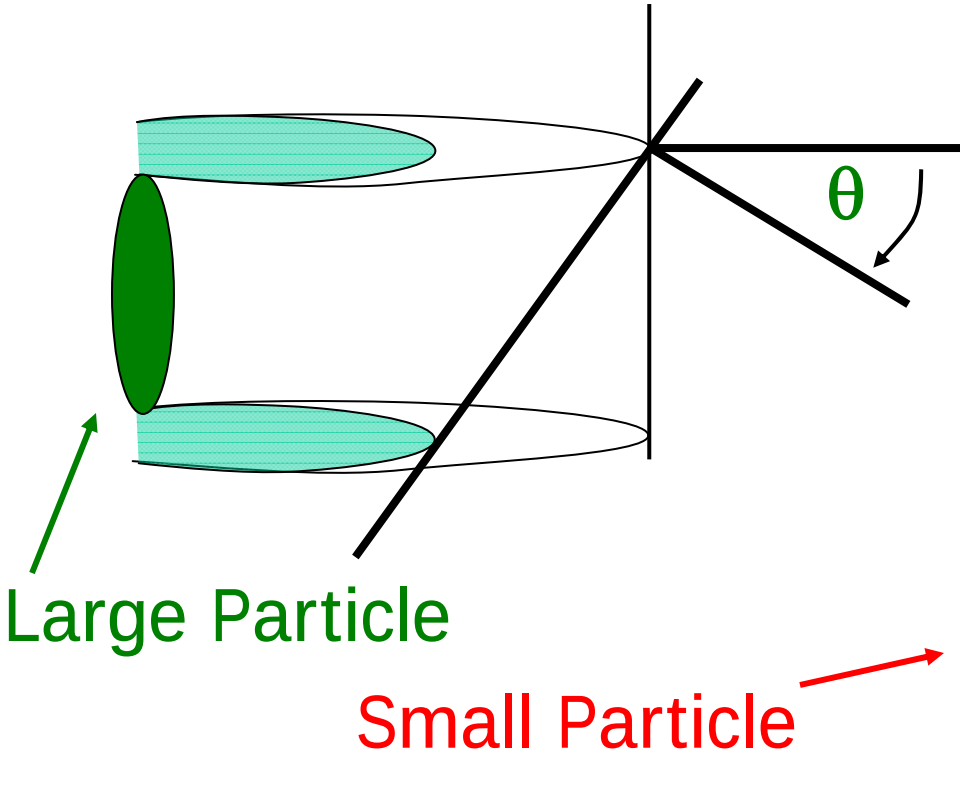
- Light Scatter occurs whether from a slit, a pinhole or a particle. It occurs at the edge of an object. A SLIT and PARTICLE of the same size produce the same diffraction pattern



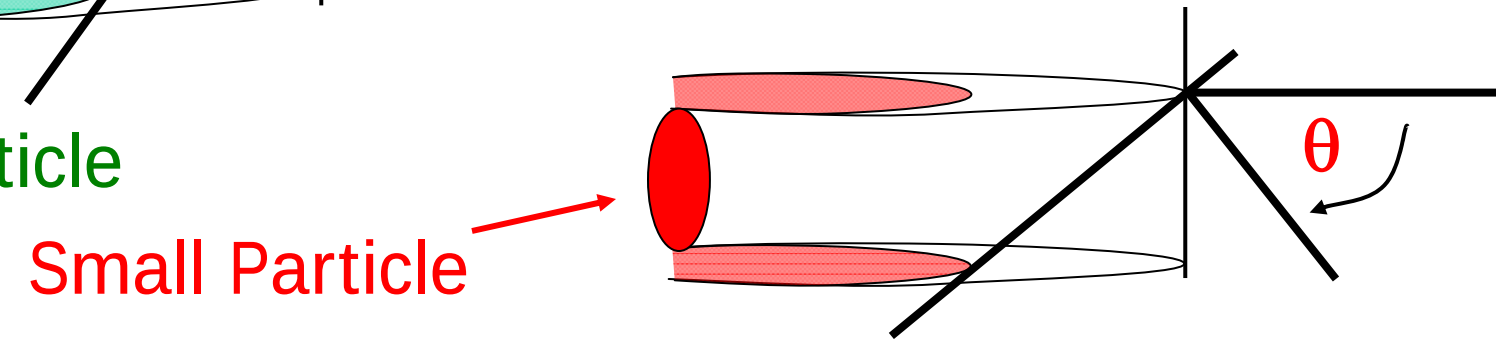


Size affects angle

- Large particles scatter light through SMALLER angles

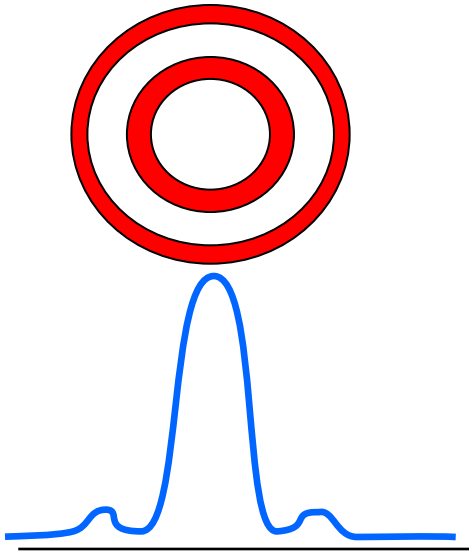


- Small particles scatter light through LARGER angles

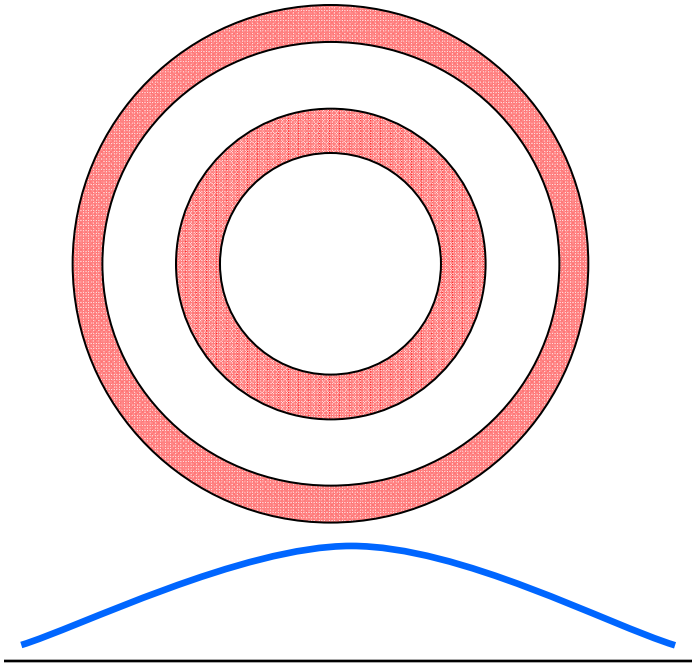


Size affects intensity

- LARGE PARTICLE:
  - Low angle scatter
  - Large signal



Narrow Pattern - High intensity



Wide Pattern - Low intensity

- SMALL PARTICLE:
  - High Angle Scatter
  - Small Signal

Other factors

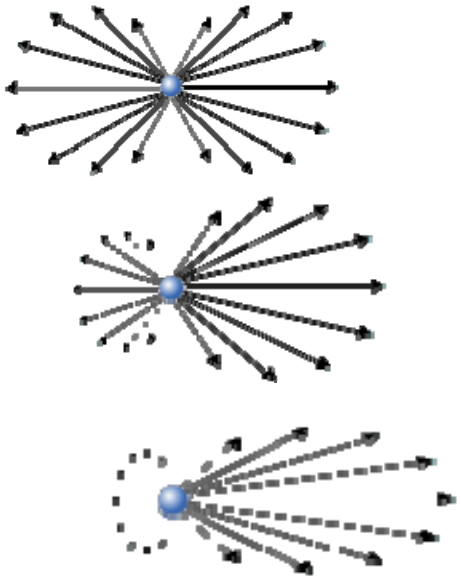
- Size, Shape, and Optical Properties also affect the angle and intensity of scattered light
- Extremely difficult to extract shape information without a priori knowledge
  - Assume hard sphere model
- Optical properties (refractive index) explain refraction
  - Key difference between Fraunhofer and Mie

Plan of Attack

- How does it work?
- What can diffraction do?
- How does it help you?
- Strong points, weak spots
- Q&A

Most flexible size analysis

- The core relationship between size and scattered light works for:
  - Nanoparticles
  - Small micronized particles
  - Large micronized particles



Wide dynamic size range

Most flexible size analysis

- The core relationship between size and scattered light works for:
  - Suspensions – solid-liquid
  - Powders – solid-gas
  - Emulsions – liquid-liquid

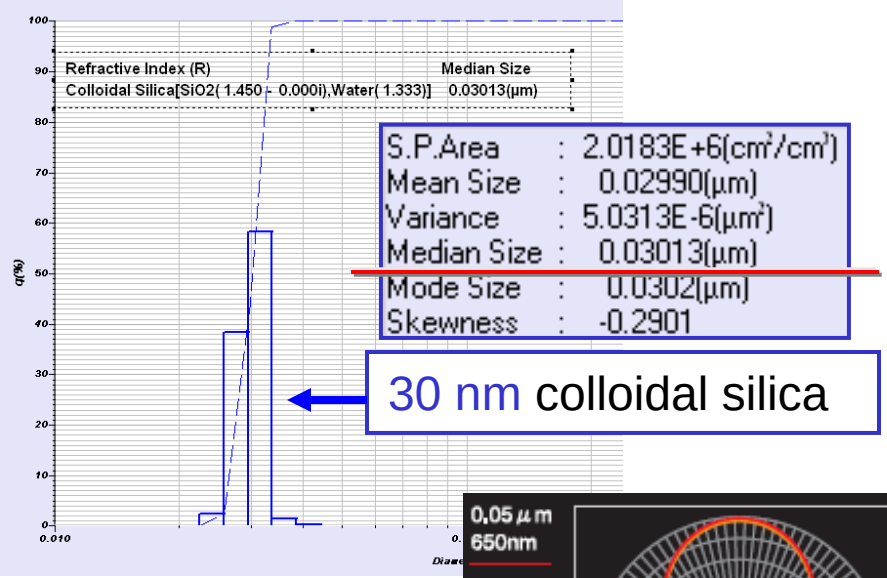
Flexible sample types

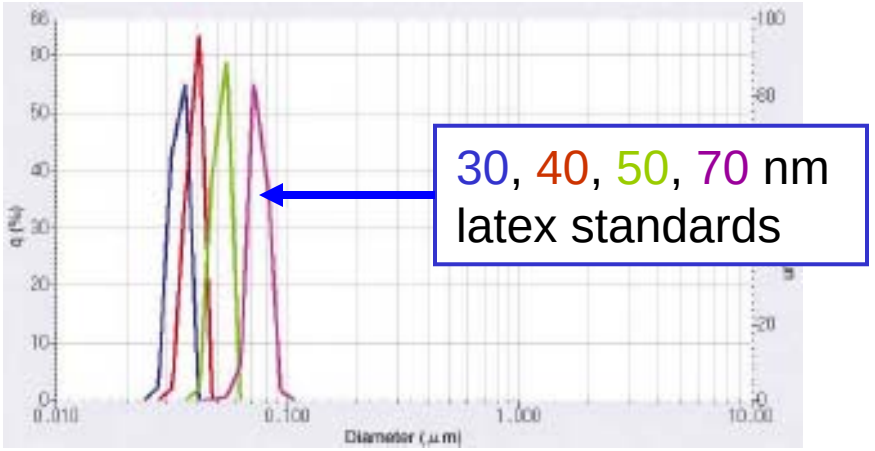
Most flexible sizing

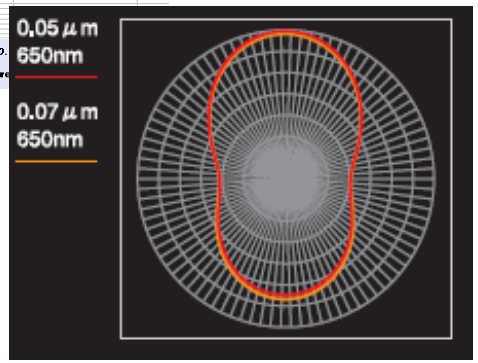
- Wide dynamic size range
- Multiple sample types
- No a priori information needed to monitor size change
- Accuracy is improved with refractive index (when smaller than approx. 30 microns)
- Very fast measurement → think seconds, not minutes
- Easy to use, easy to interpret
- First principal measurement → no calibration

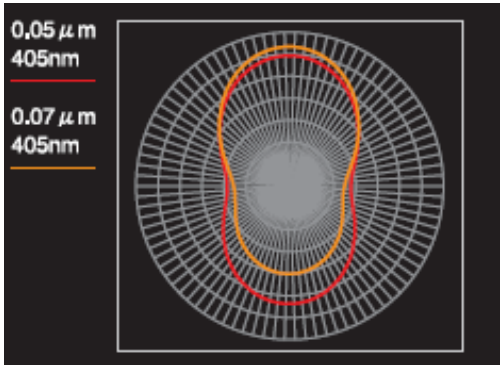
Nanoparticle Sensitivity

- Laser diffraction can measure 30 nm!
  - Typical setup is 2 light sources: red & blue

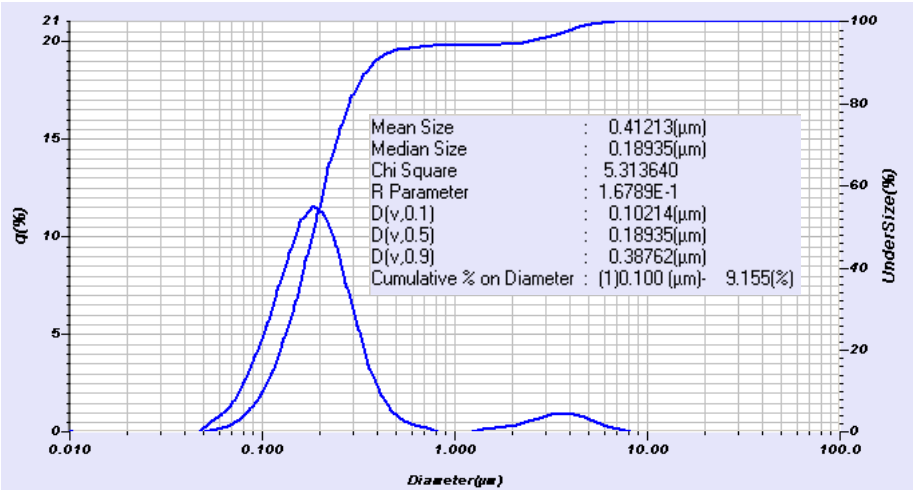


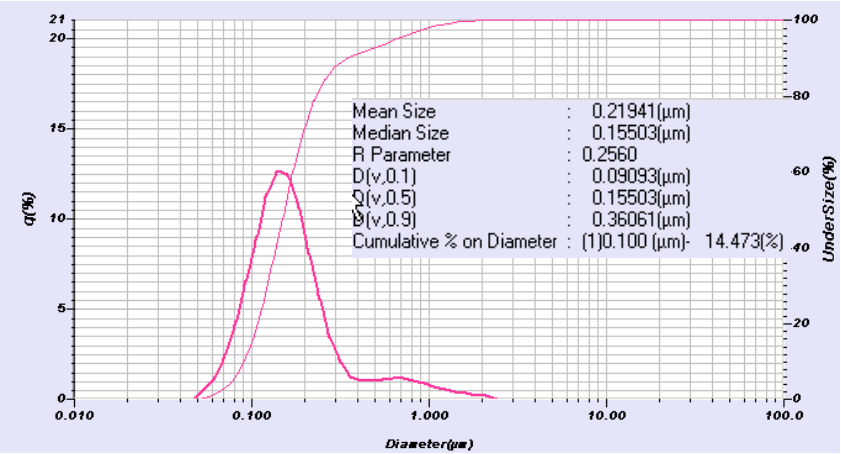






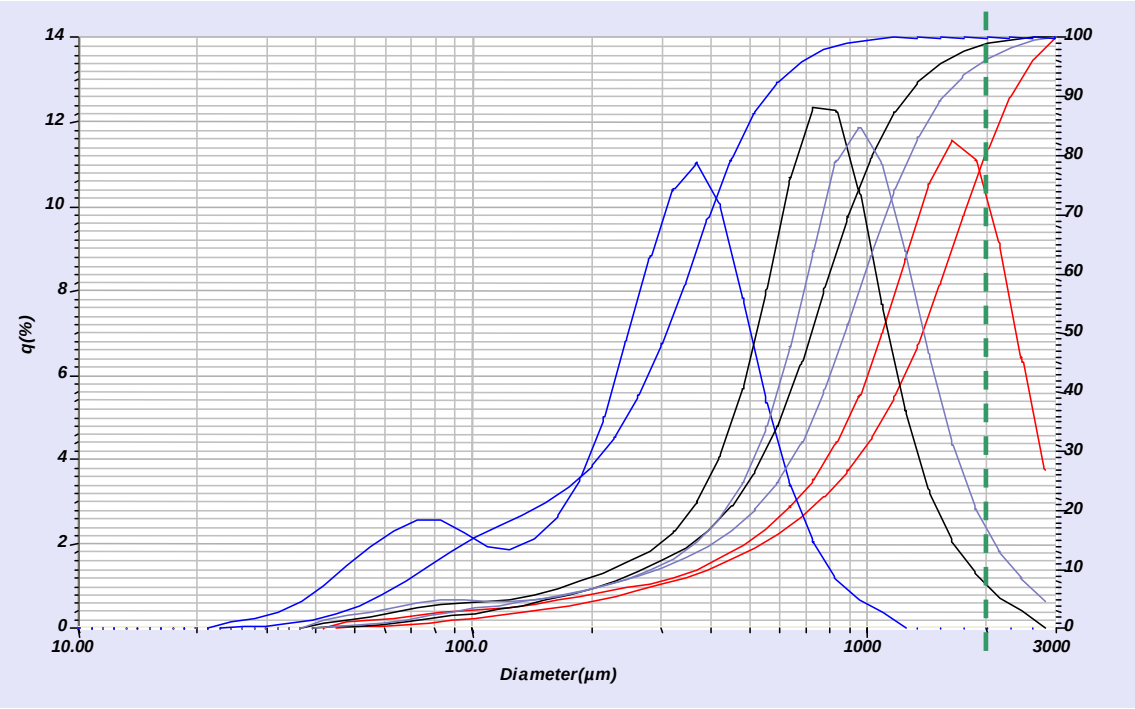
Nanoparticle Sensitivity



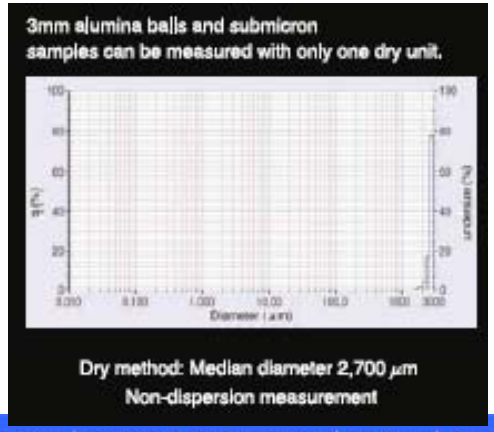


- Some (unfounded?) concerns with particles < 100 nm
- Diffraction good at determining sub-100 nm particles in presence of larger particles
- Software set to display % under any given size
- Data shown left is for skin cream and TiO2 suspension

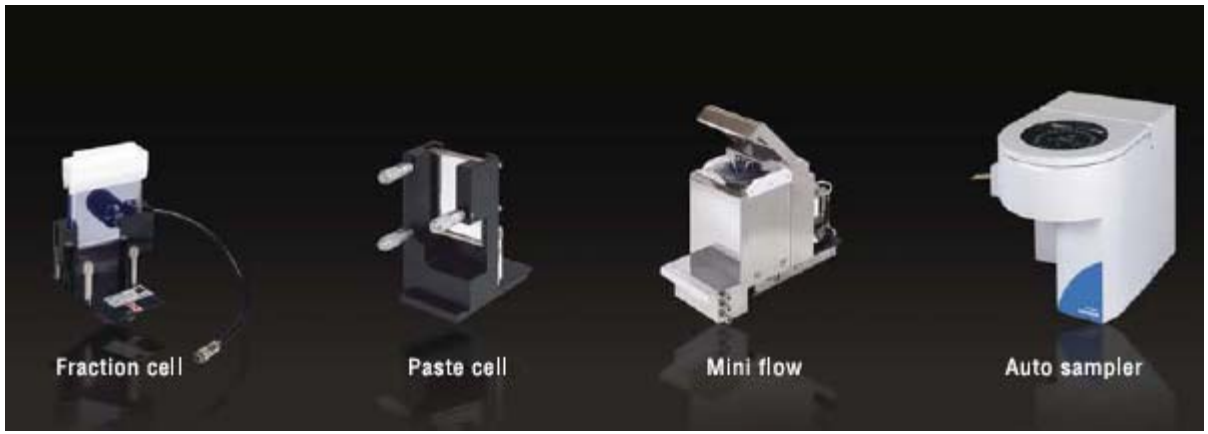
Large Particle Sensitivity



Coffee Results



Flexible Sample Handlers



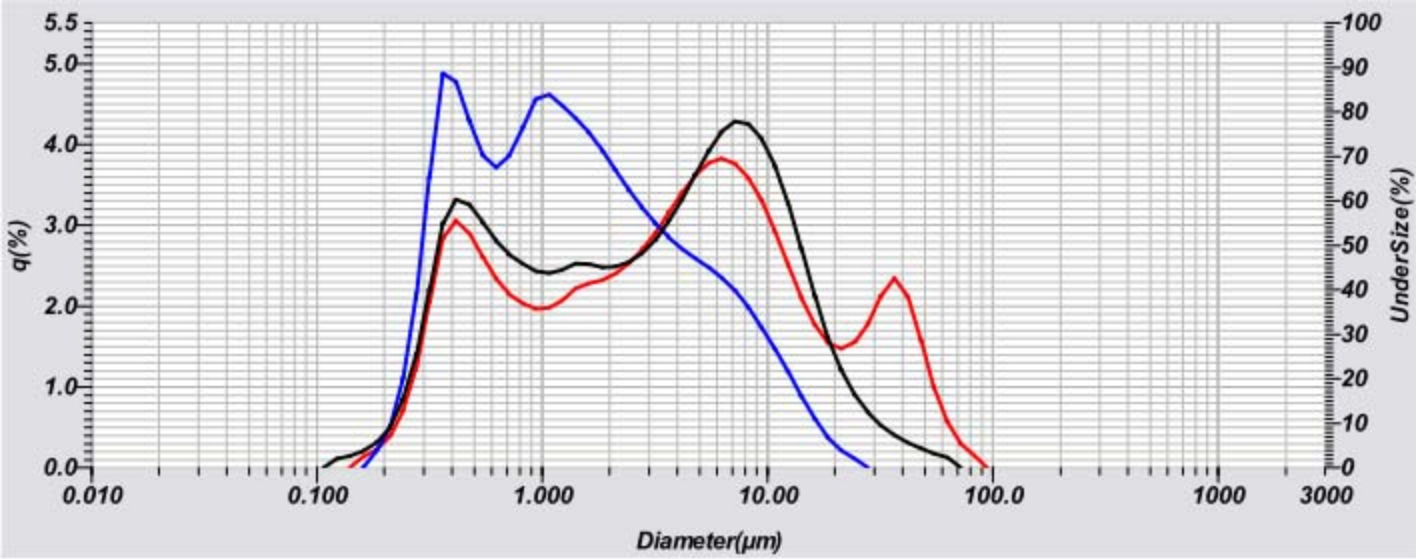
10 ml 35 ml 200 ml powders

- Wide range of sample cells depending on application
- High sensitivity keeps sample requirements at minimum
- Technology has advanced to remove trade-offs

Plan of Attack

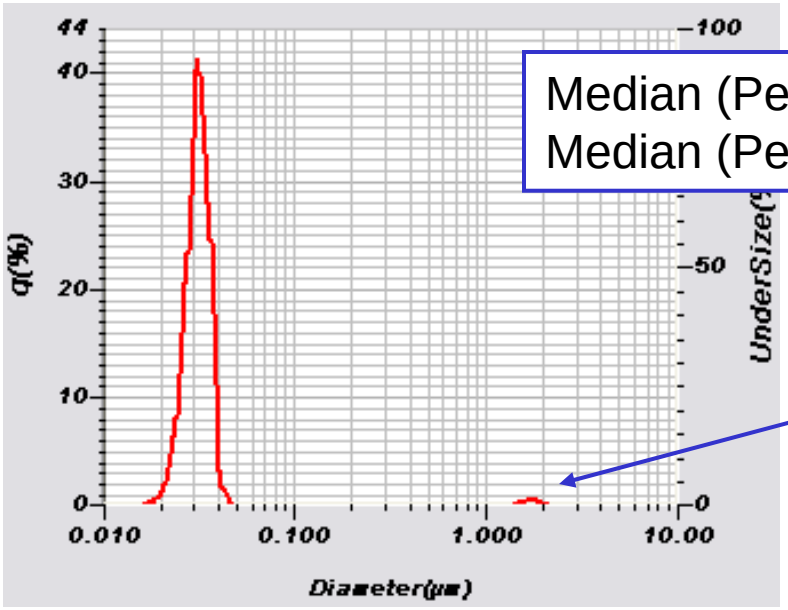
- How does it work?
- What can diffraction do?
- How does it help you?
- Strong points, weak spots
- Q&A

Monitor Process Quality



- **Black** size distribution is internal standard
- **Red** result is considered a passing batch
- **Blue** result is a bad batch
- Highlights problem in production
- All accomplished with complex product formulation

Monitor Product Quality

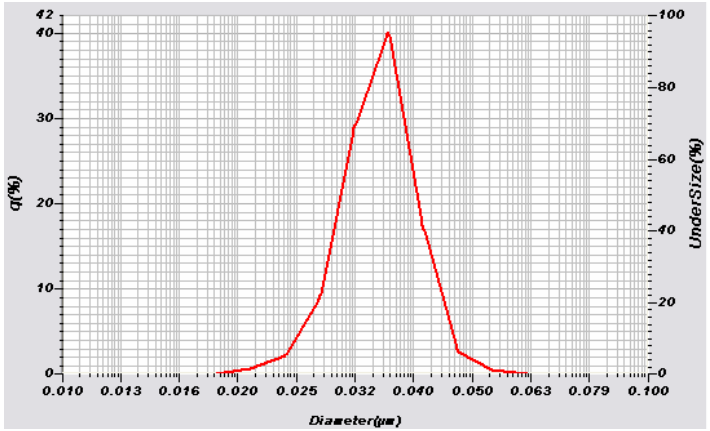


Median (Peak 1) : 0.031 μm
Median (Peak 2) : 1.75 μm

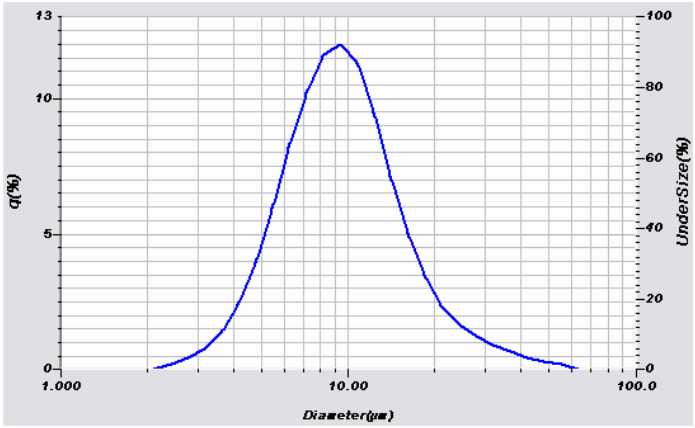
0.05wt%

- Product quality and performance can be located at tails
- Diffraction is “resolution limited” technique, but can still have success finding outlier populations

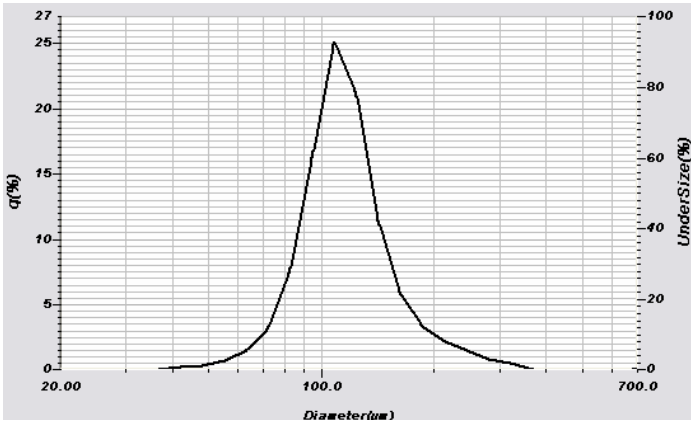
Minimize Sample (MiniFlow)



Colloidal Silica (weak scatterer)
Median (D50): **35 nm**
Sample Amount: **132 mg**



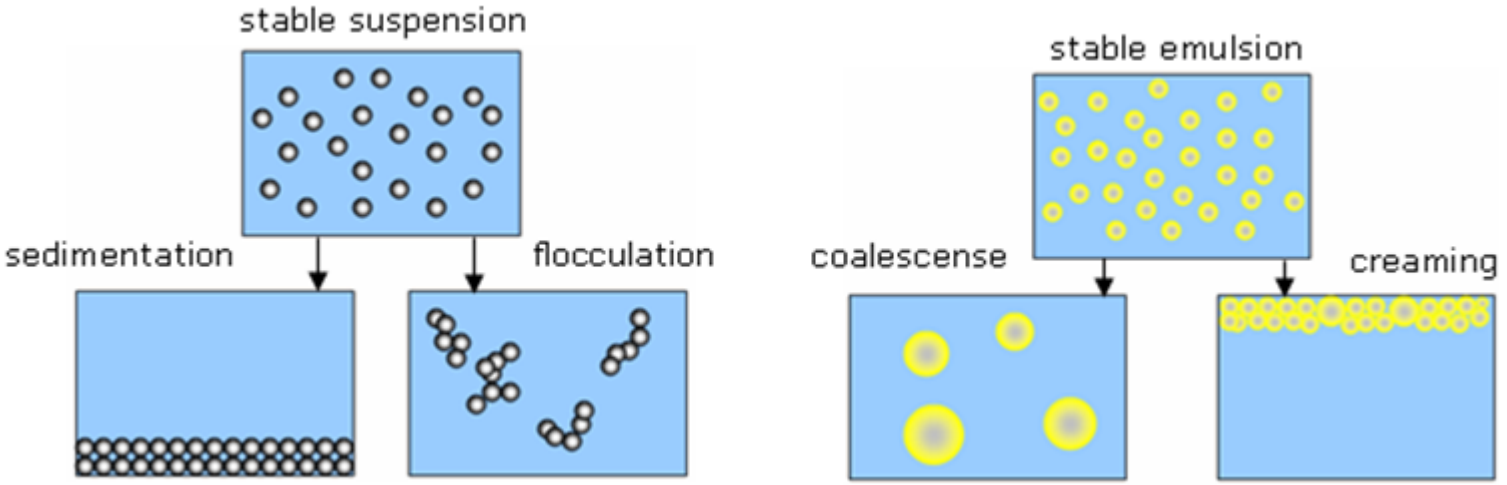
Magnesium Stearate
Median (D50): **9.33 μm**
Sample Amount: **0.165 mg**



Bio-degradable Polymer
Median (D50): **114 μm**
Sample Amount: **1.29 mg**

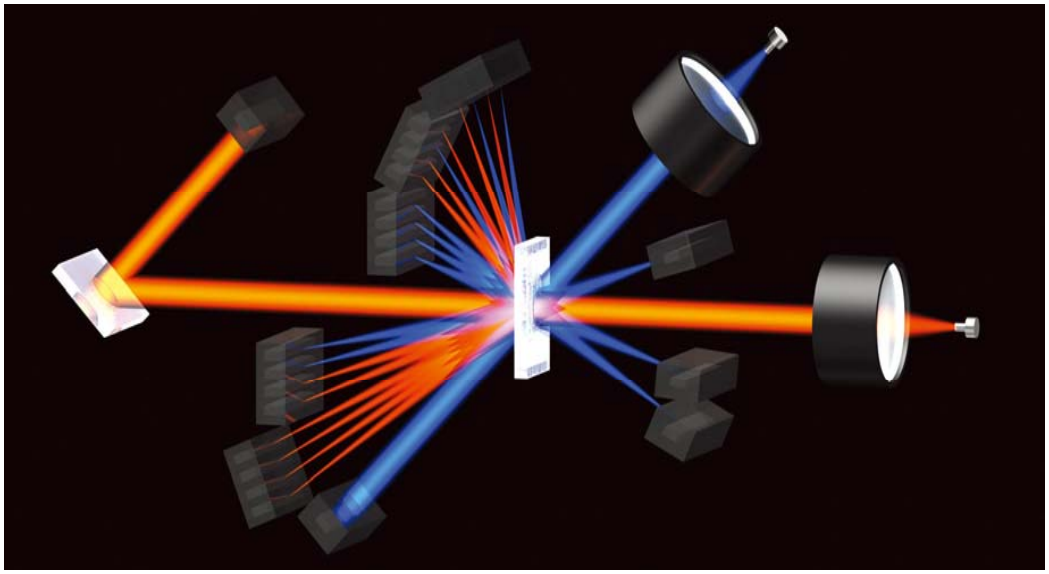
Measurement Workflow

- Prepare the sample
  - Good sampling and dispersion a must!
  - May need to use surfactant or admixture



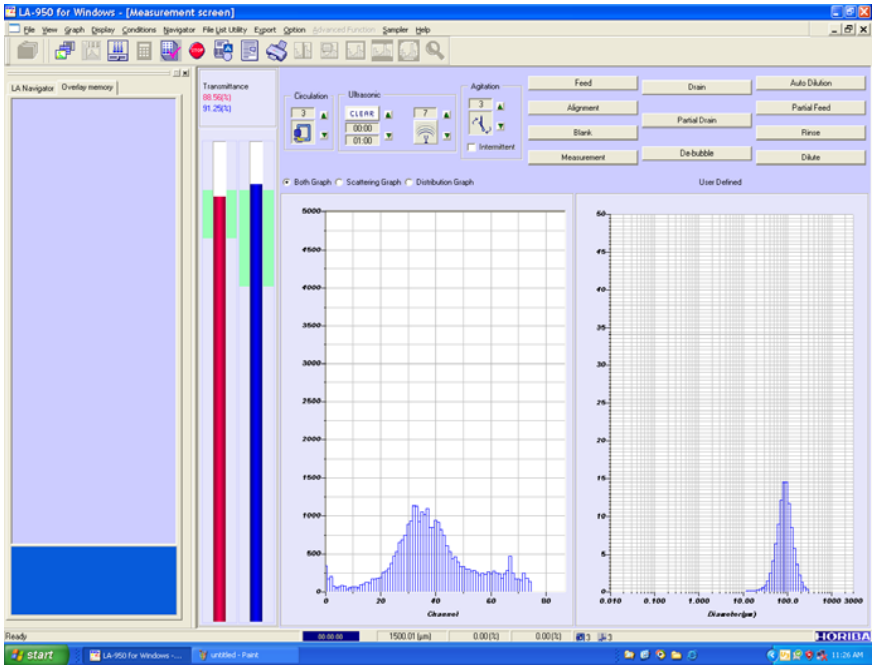
Measurement Workflow

- Prepare the system
  - Align laser to maximize signal-to-noise
  - Acquire blank/background to reduce noise

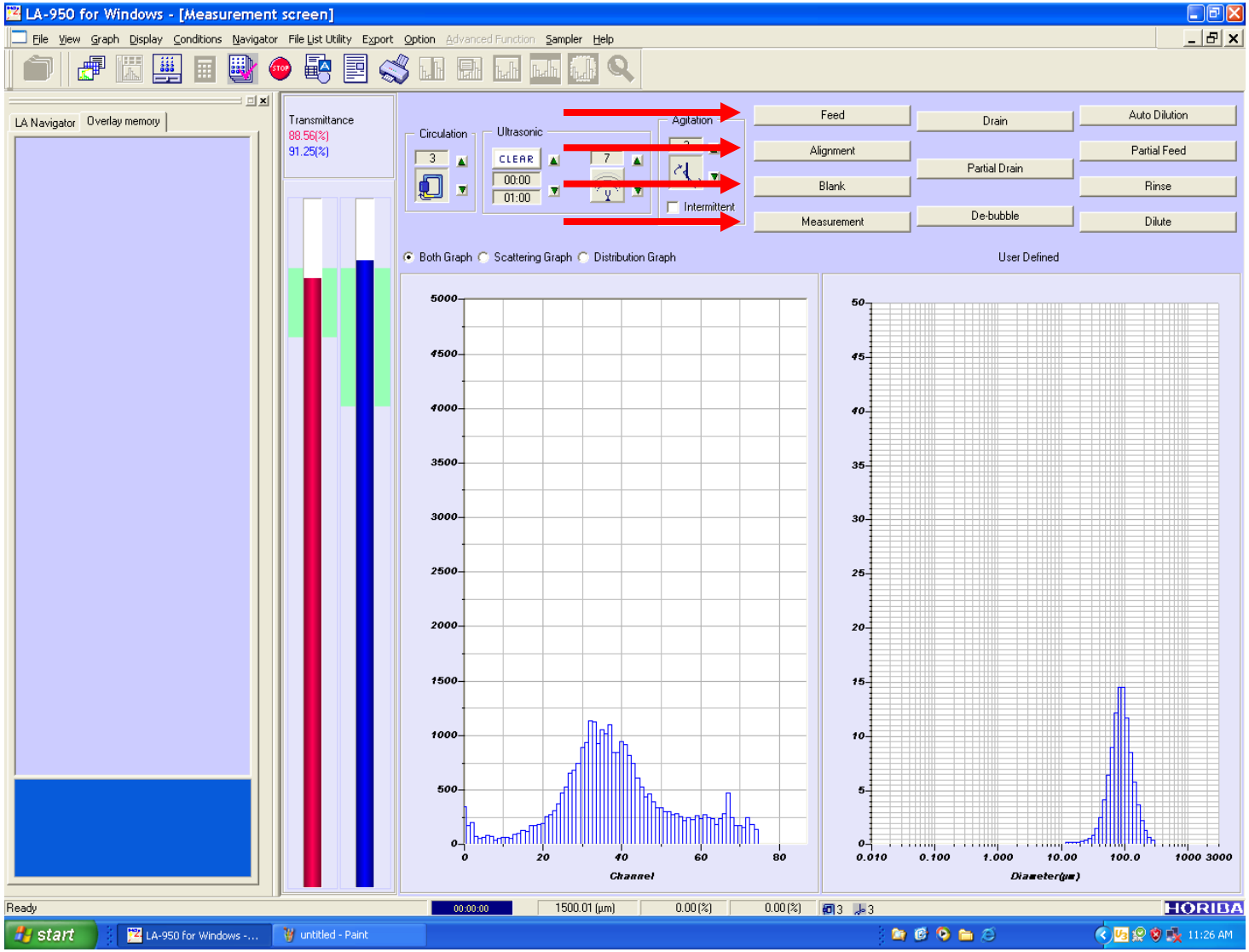


Measurement Workflow

- Introduce sample
  - Add sample to specific concentration range
  - Pump sample through measurement zone
  - Final dispersion (ultrasonic)

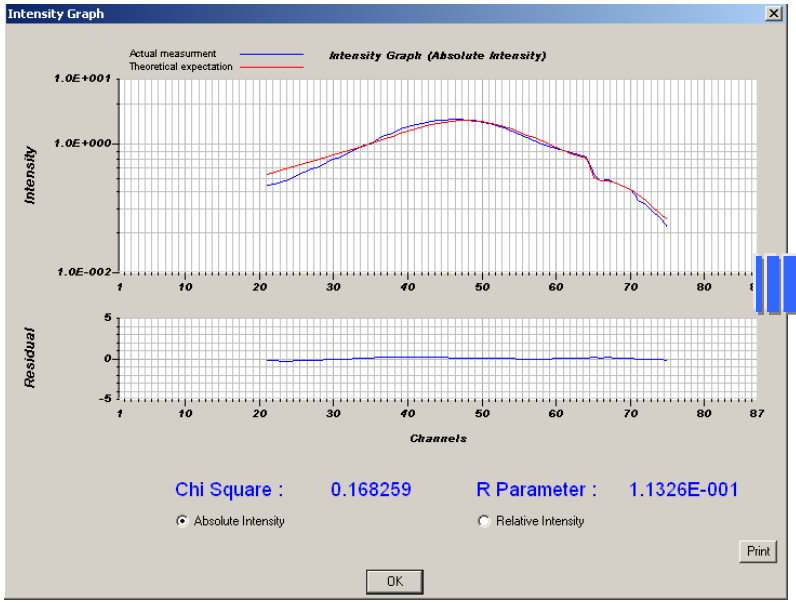


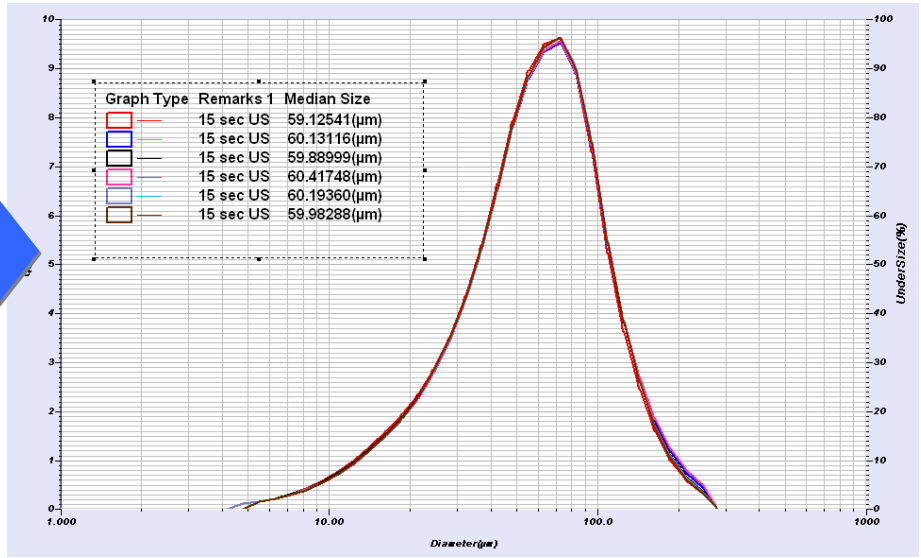
Measurement Workflow



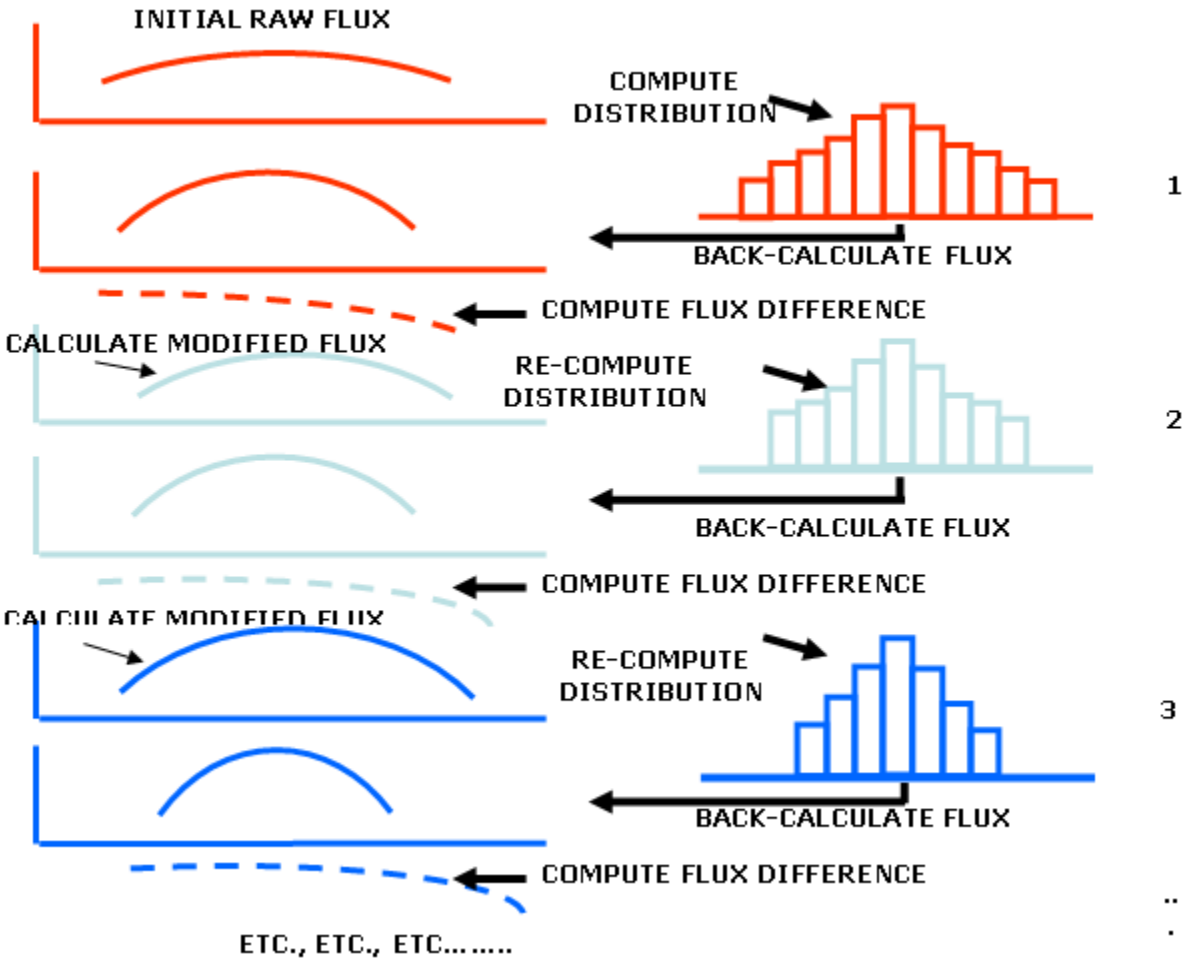
Measurement Workflow

- Measurement
  - Click “Measure” button
    - *Hardware* measures scattered light distribution
    - *Software* then calculates size distribution



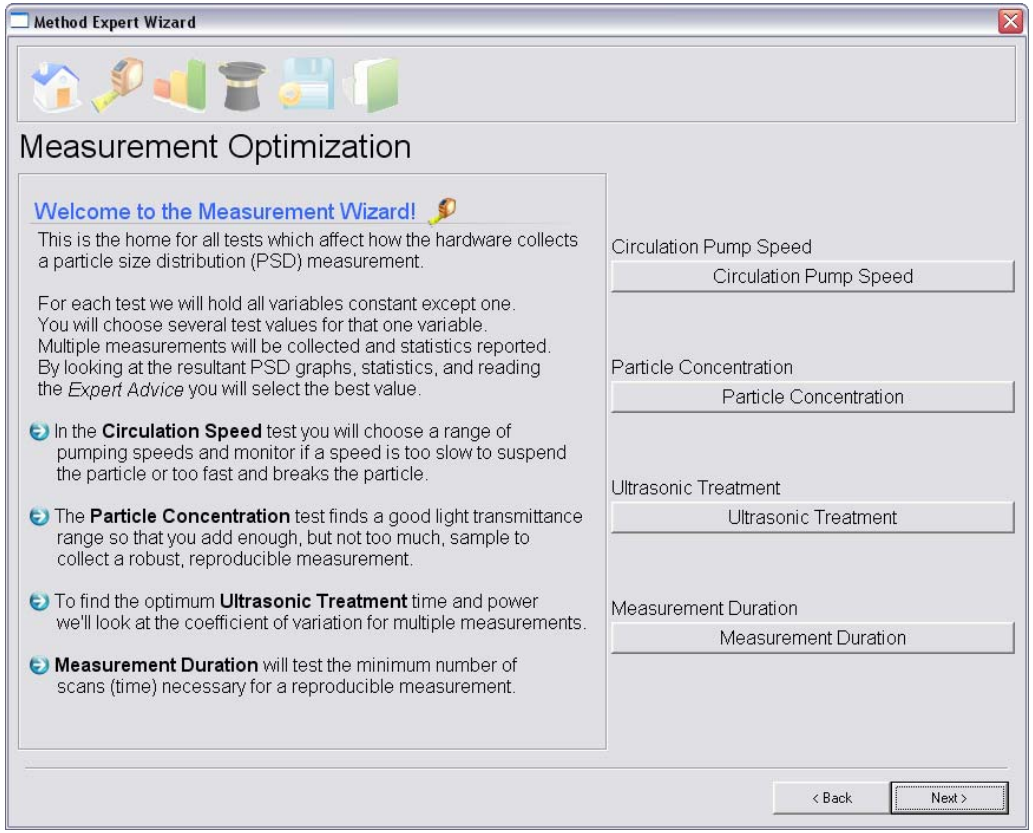


Iterative Calculation



LA-950 Method Expert

There are four important tests…



Circulation

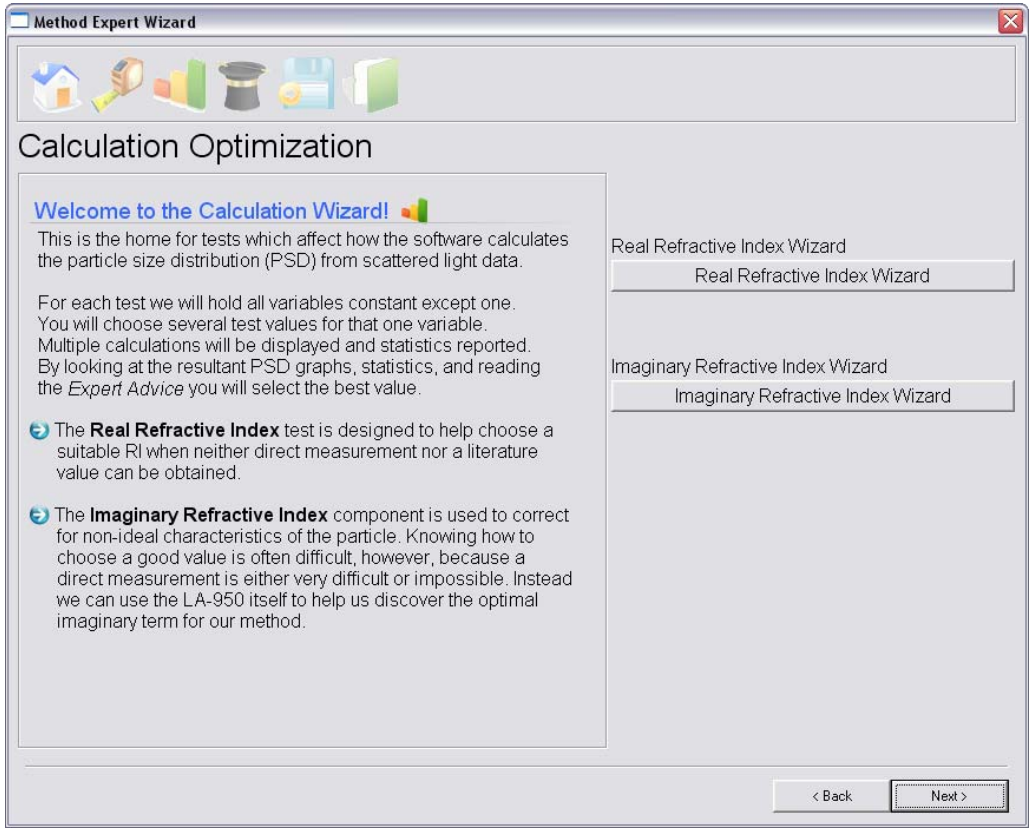
Concentration

Dispersion

Duration

LA-950 Method Expert

There are two important tests…



Real RI

Imaginary RI

LA-950 Method Expert

Why is the test important?

What does the test do?

How will the results be displayed?

What is the best value?

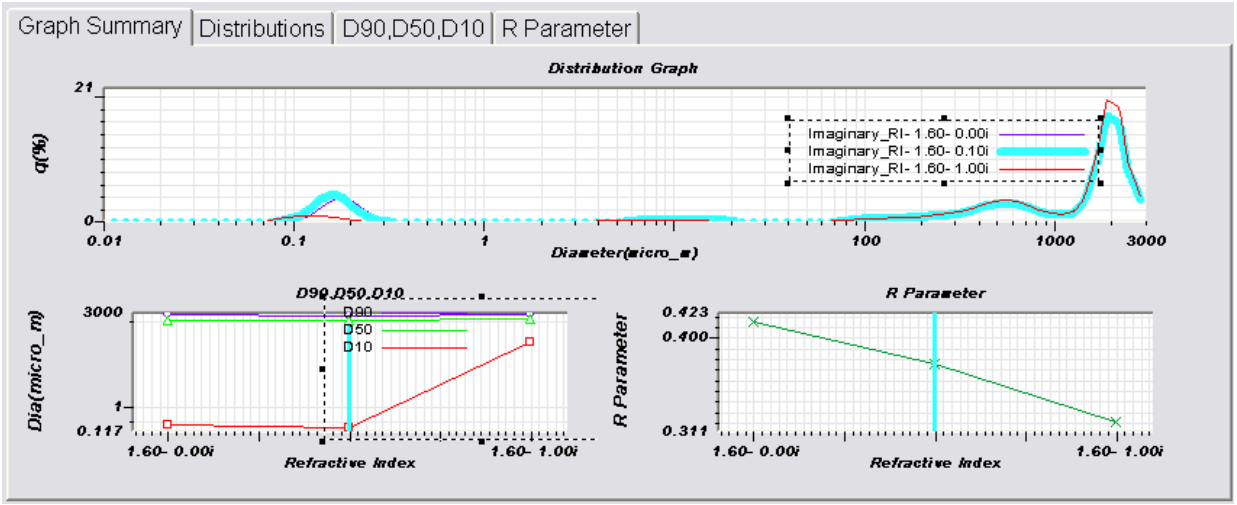
User selects up to 5 values for testing

LA-950 Method Expert

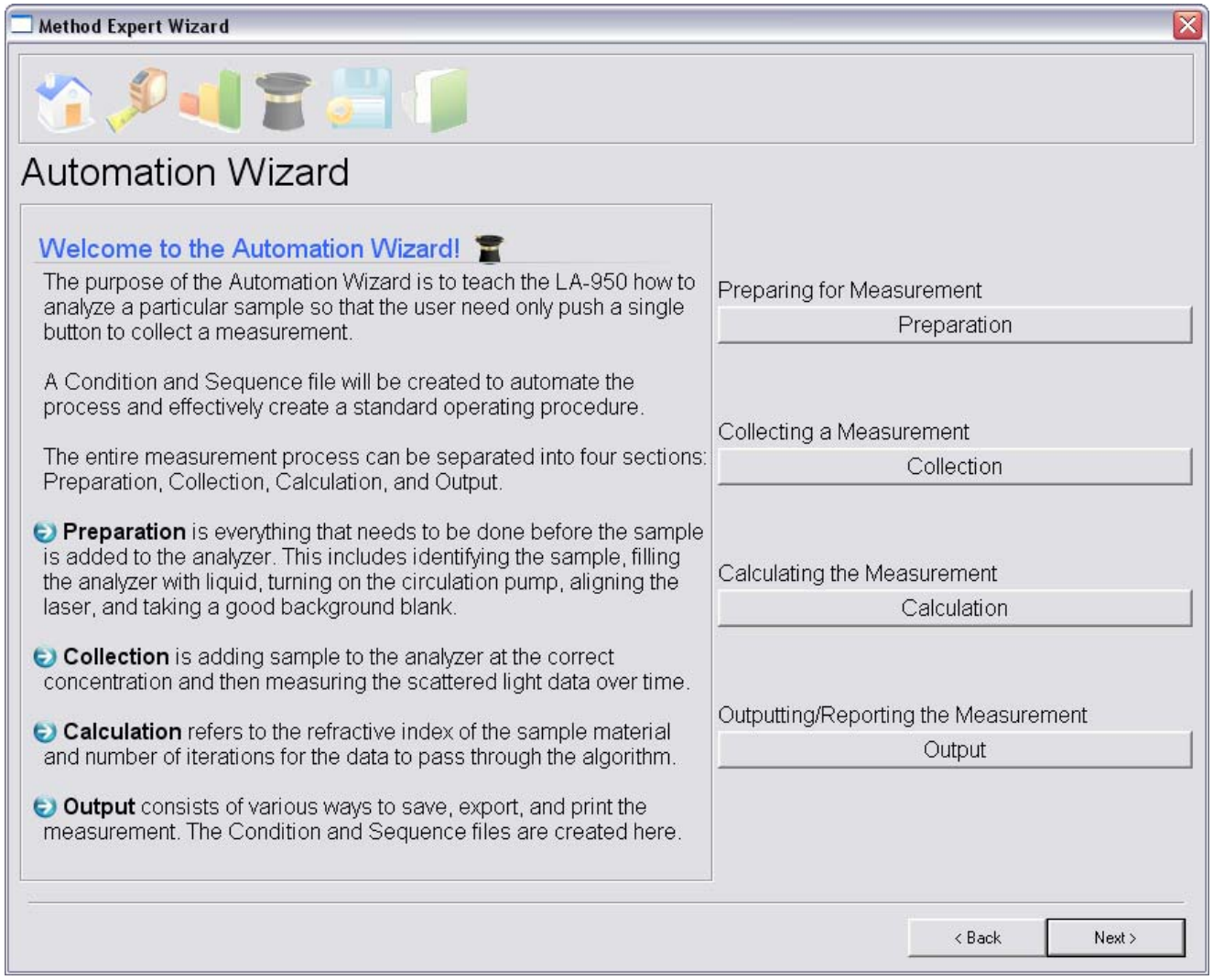
Method Expert guides user to prepare the LA-950 for each test

Results displayed in multiple formats:

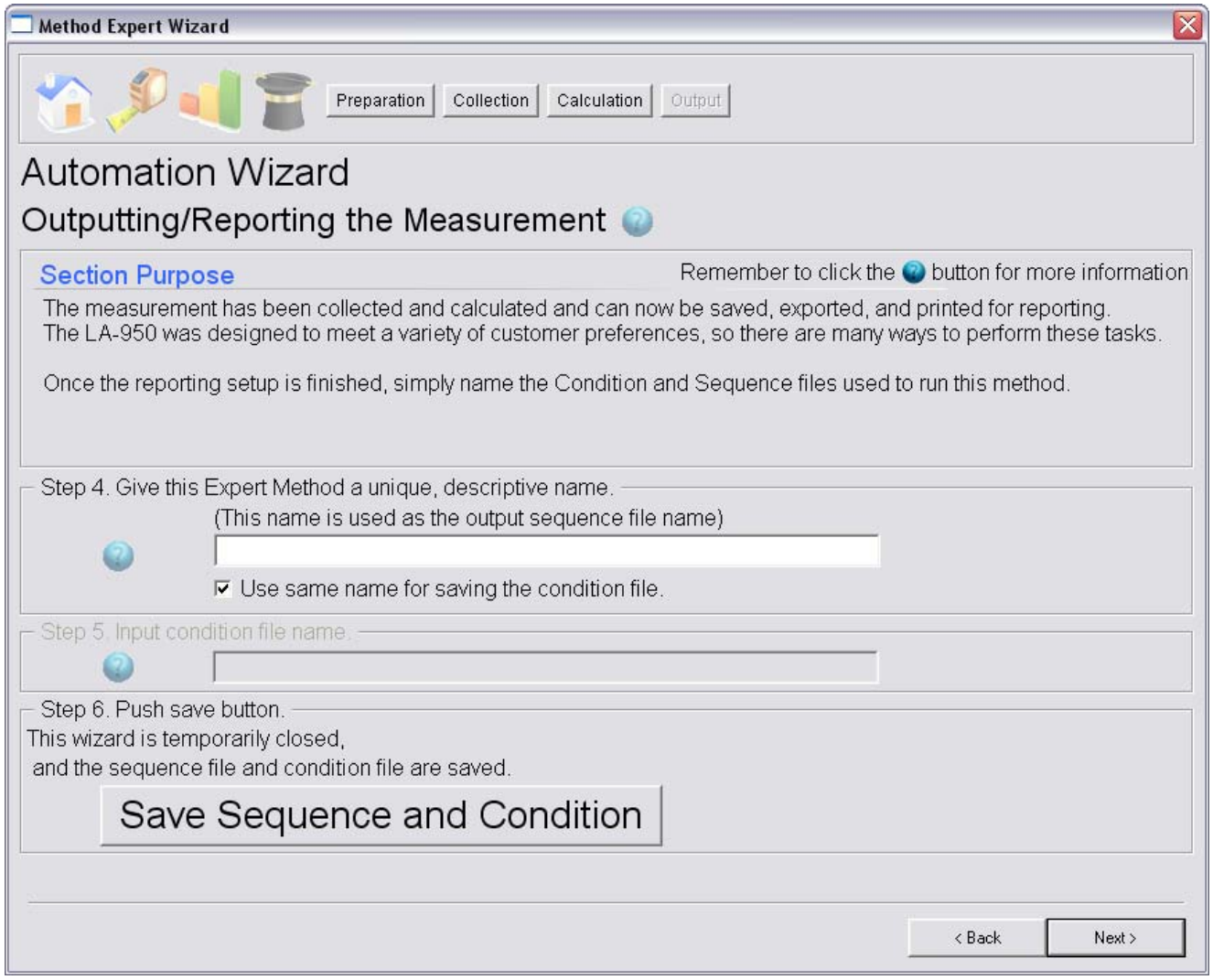
PSD, D50, R-parameter



LA-950 Method Expert



LA-950 Method Expert



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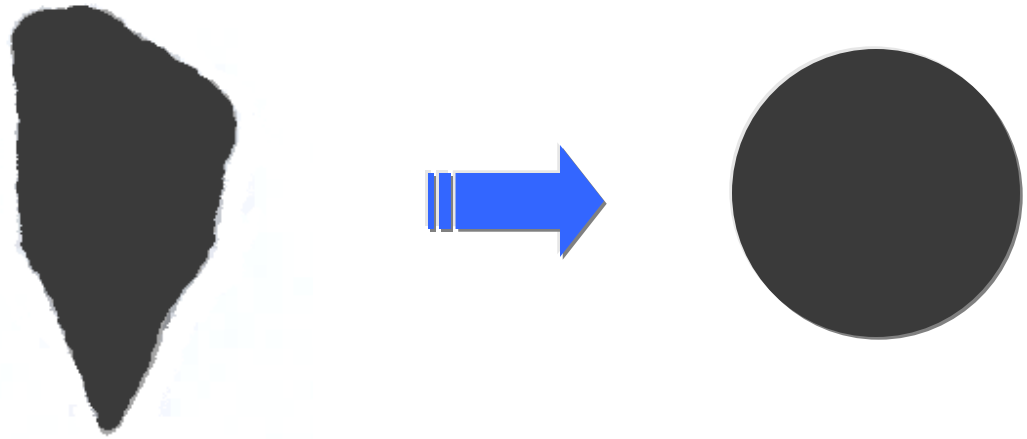
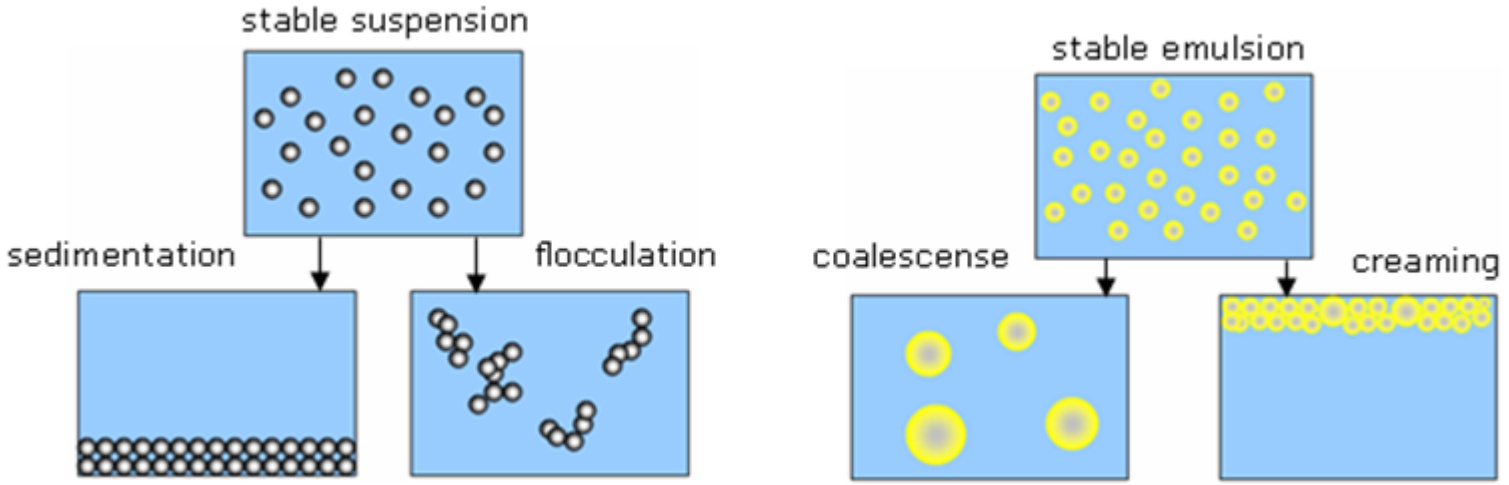
Diffraction Drawbacks

- Assumes hard spherical model
  - Most materials are aspherical
  - Error is introduced
  - Data cannot be directly compared to other techniques measuring different property



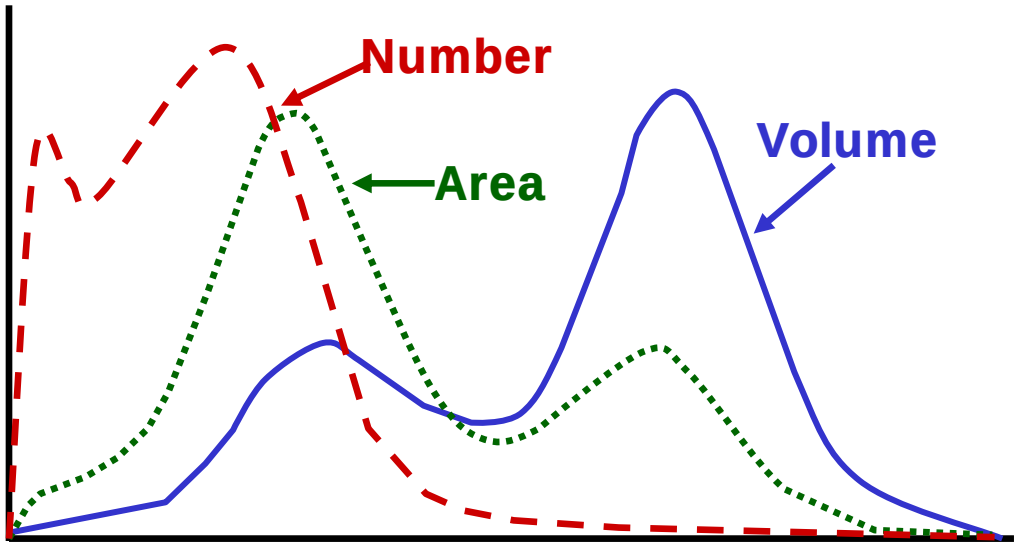
Diffraction Drawbacks

- "Optical" concentration range
  - Dilution can cause stability issues
  - Especially difficult for creams and pastes



Diffraction Drawbacks

- Volume basis by default
  - Although excellent for mass balancing, cannot calculate number basis without significant error



The Benefits

- Wide size range
  - Most advanced analyzer measures from 30 *nano* to 3 *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 establishment

Plan of Attack

- How does it work?
- What can diffraction do?
- How does it help you?
- Strong points, weak spots
- Q&A

For More Information

ISO 13320:2009 – the go-to guidance resource
Particle Size Measurements; Fundamentals, Practice, Quality by Henk Merkus

Visit www.horiba.com/us/particle
Previously recorded webinars:
-- Laser Diffraction Performance
-- LA-950 Method Expert Software
-- Understanding and Interpreting Your PSA Results
-- Setting Attainable Size Specifications
-- "Boot Camp" Laser Diffraction 6-part Training Course
Many application and technical notes

Ask a question at labinfo@horiba.com

Keep reading the monthly HORIBA Particle e-mail newsletter!

Visit the Download Center to find the video and slides from this webinar.

Thank-you