Materials Characterization Workshop – Determination of Particle Size Distribution of Powders by Various Analytical Methods, Including Characterization of Particle Shape

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Outline

- Particle Sizing
  - Sedimentation
  - Light Scattering – Static and Dynamic
  - Electrical Sensing Zone
  - Air Permeability
- Particle Shape Evaluation

Particle Size Distribution Analysis

What is needed from Particle Size Distribution Analyses?

- Repeatability
- Reproducibility
- Reliability
- High throughput
- Prediction of behavior

X-ray Gravity Sedimentation – SediGraph III 5120

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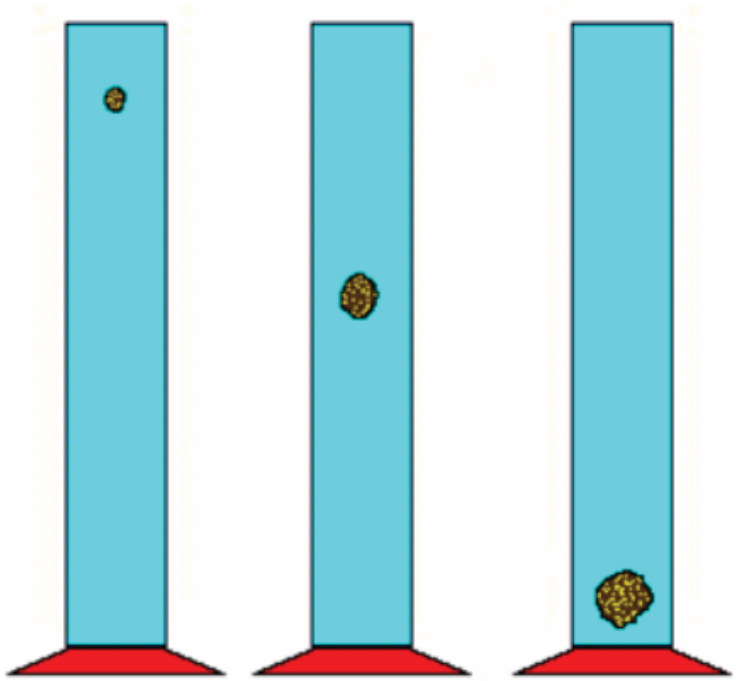
Particle Size Distribution

X-ray Gravity Sedimentation

- Based upon a classical particle sizing method – Stokes' Law
- Size determined from settling velocity
- Same principle as Andreasen Pipette
- Direct Mass concentration detection
- X-ray attenuation is proportional to mass in x-ray beam



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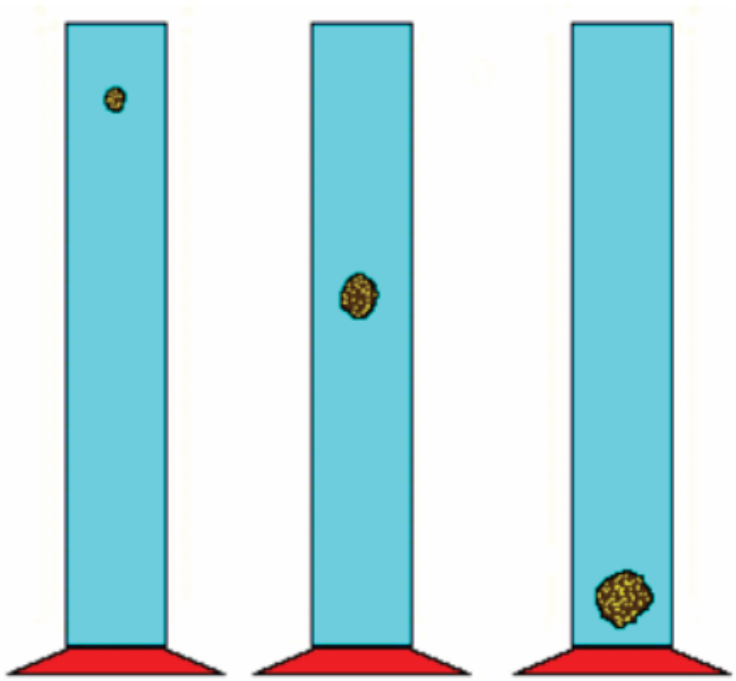

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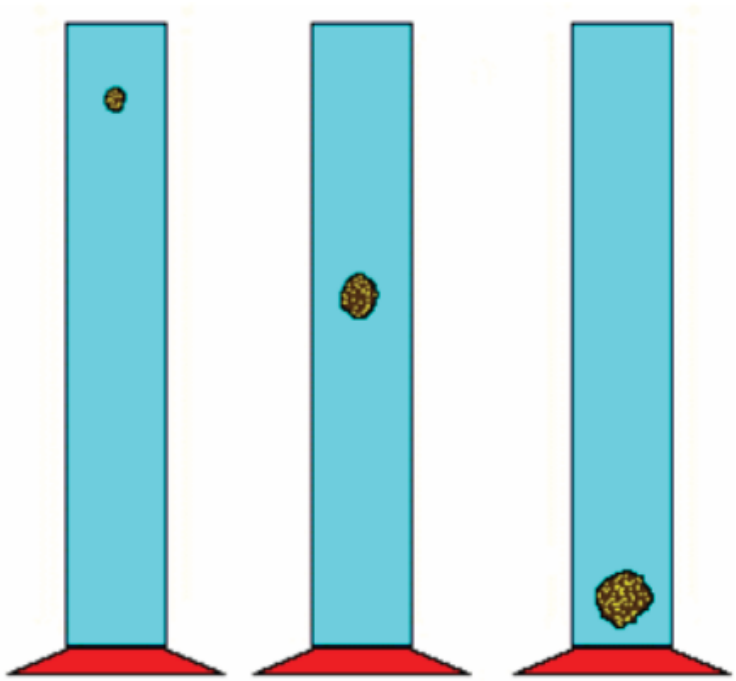

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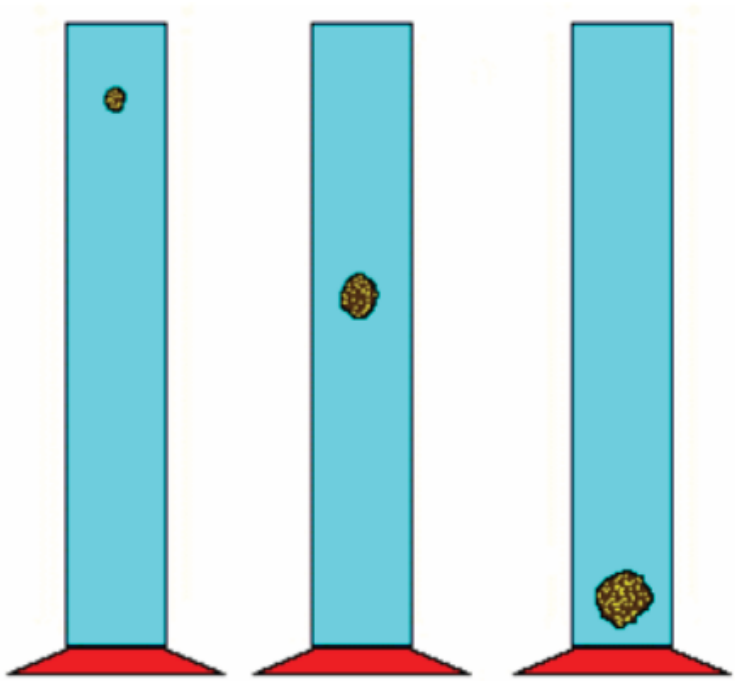

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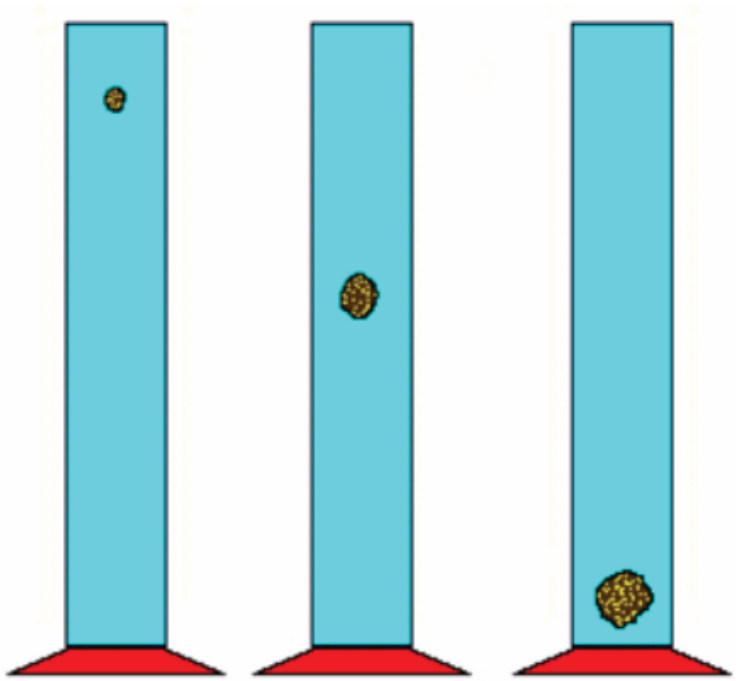

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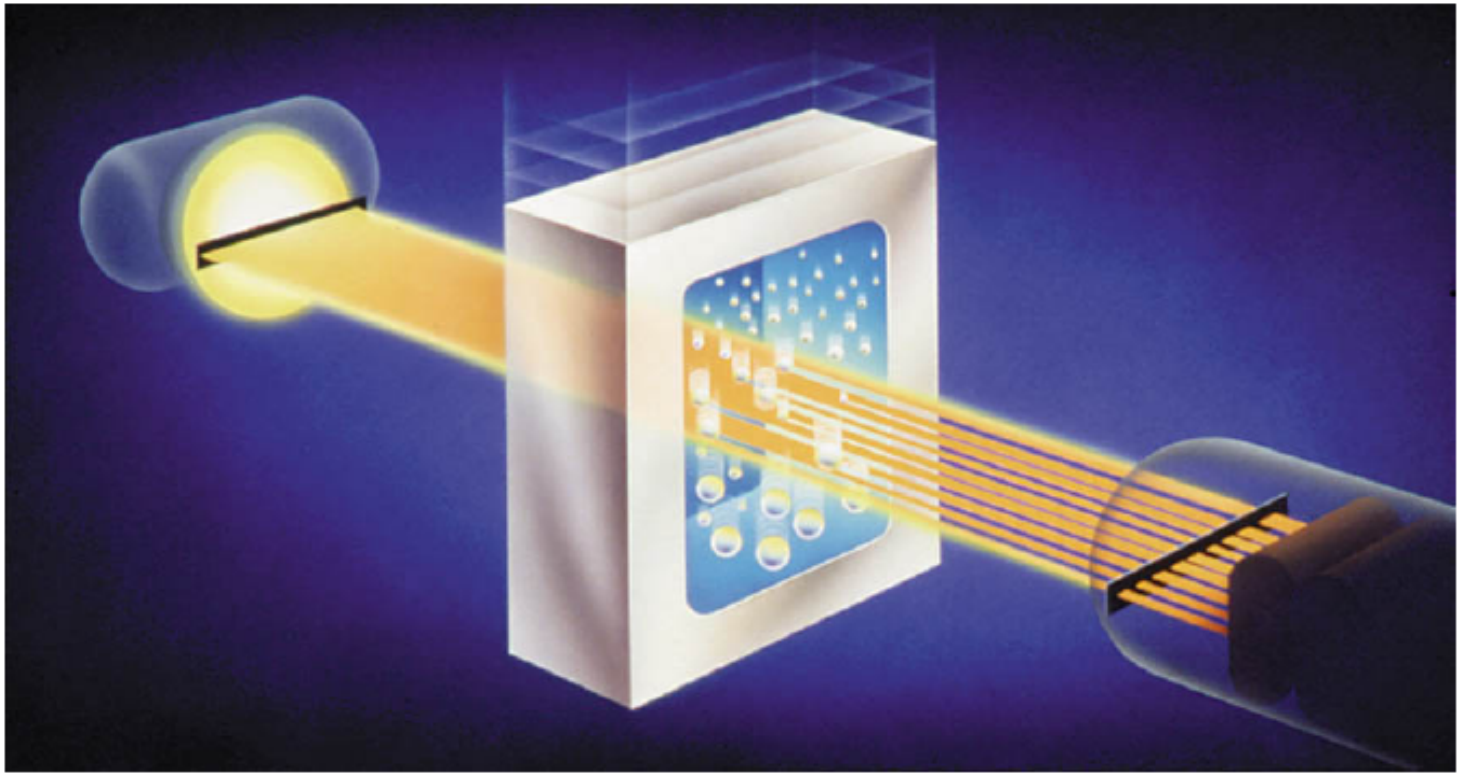

Stokes' Equation - 1891

$$D = \sqrt{\frac{18 \cdot \eta \cdot v}{(\rho - \rho_o) \cdot g}}$$

where:

- D = particle diameter
- η = liquid viscosity
- v = sedimentation velocity
- ρ = particle density
- ρ_o = liquid density
- g = acceleration due to gravity

X-ray Gravity Sedimentation

Applications

- Developed for fine particles such as kaolin
- Used extensively in the coatings, ceramics, and cement industries
- Used for calcium carbonate, talc, kaolin, titanium dioxide, pigments
- Used widely for sediments and soils
- Used in mines, pilot labs, process control labs, quality control labs
- Organic materials without heavy atoms, like chlorine for example, cannot be analyzed due to low x-ray absorption

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X-ray Gravity Sedimentation

Advantages

- Accounts for particle mass outside analysis range
- Analyzes higher concentrated slurries than most other techniques
- Provides reliable analyses of wide size range – 300 μm – 0.1 μm
- Requires only readily-available physical constants as parameters

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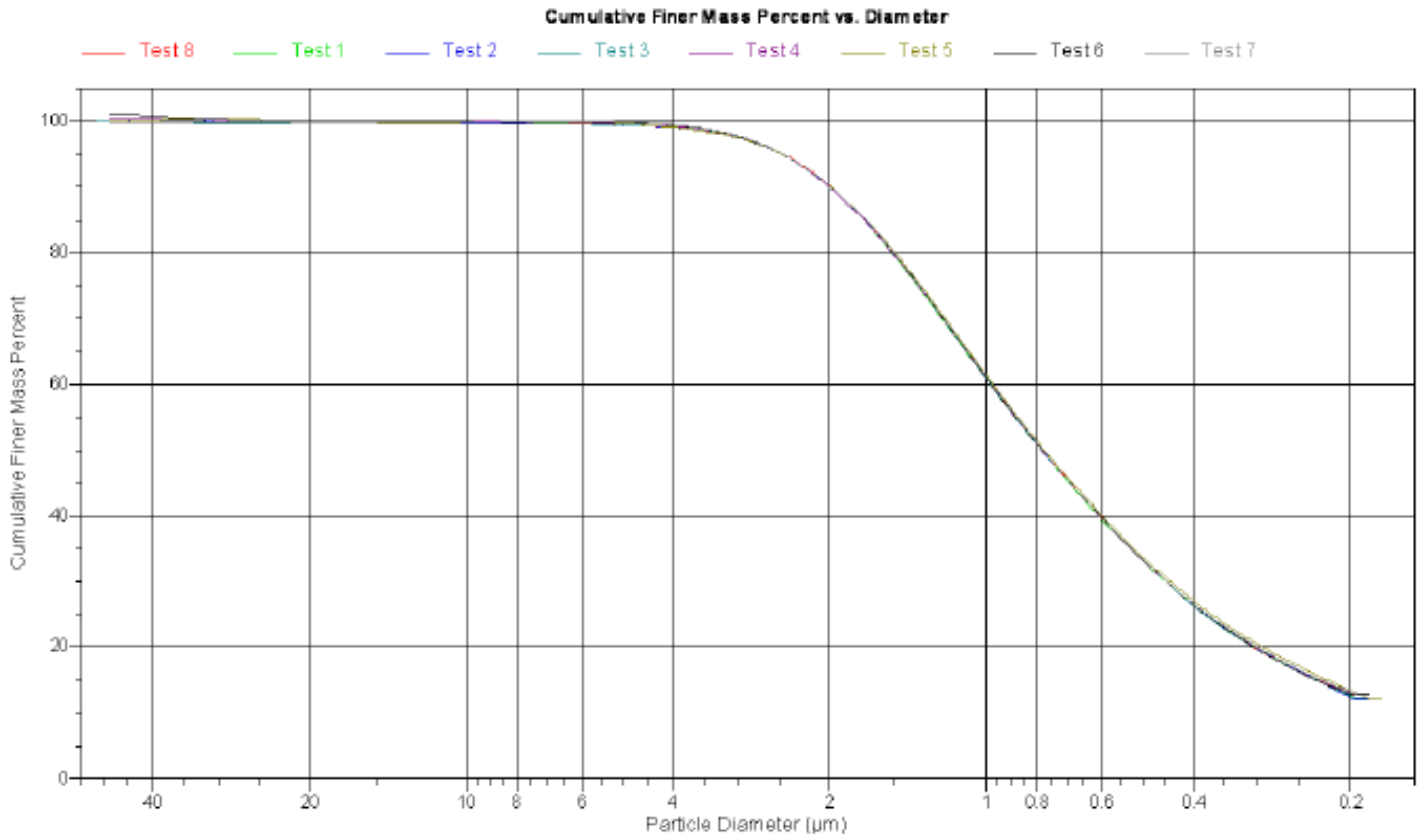
Particle Size Distribution of Fine Calcium Carbonate



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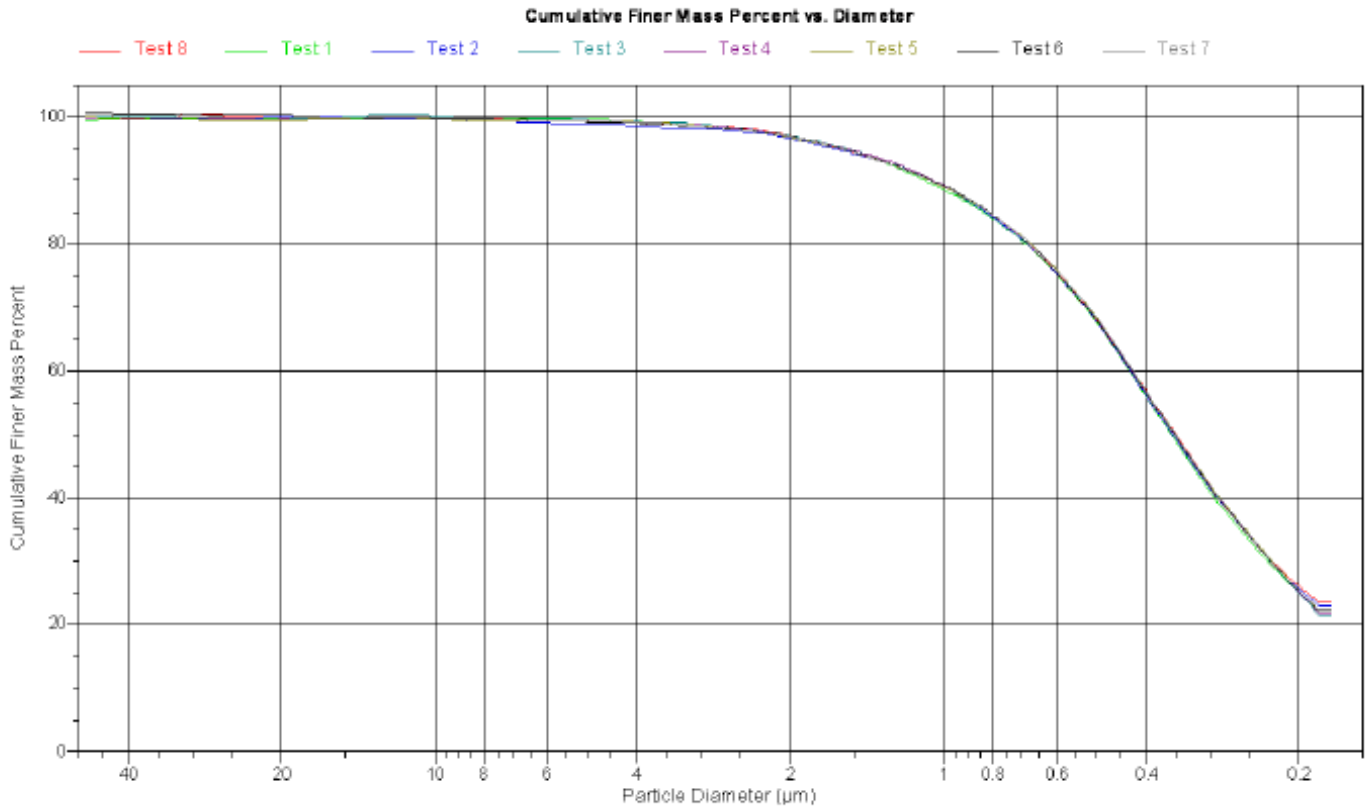
Particle Size Distribution of Kaolin



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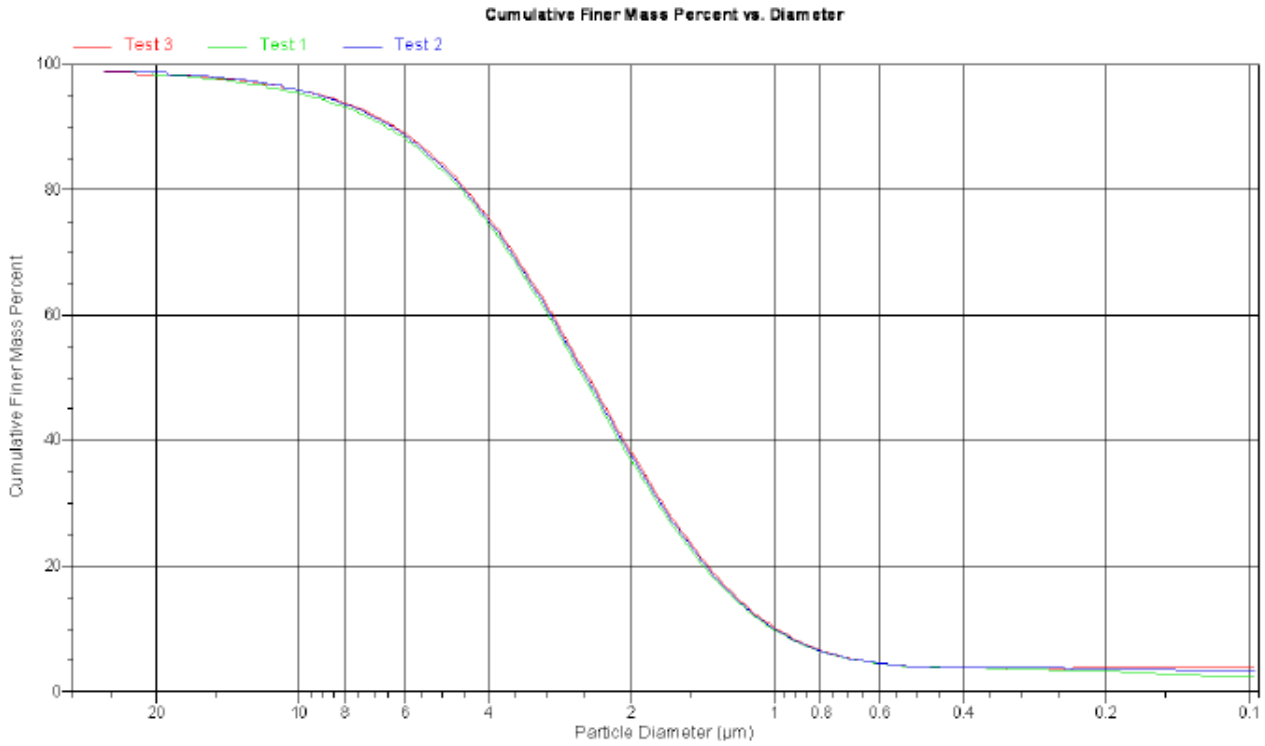
Particle Size Distribution of Tungsten Carbide



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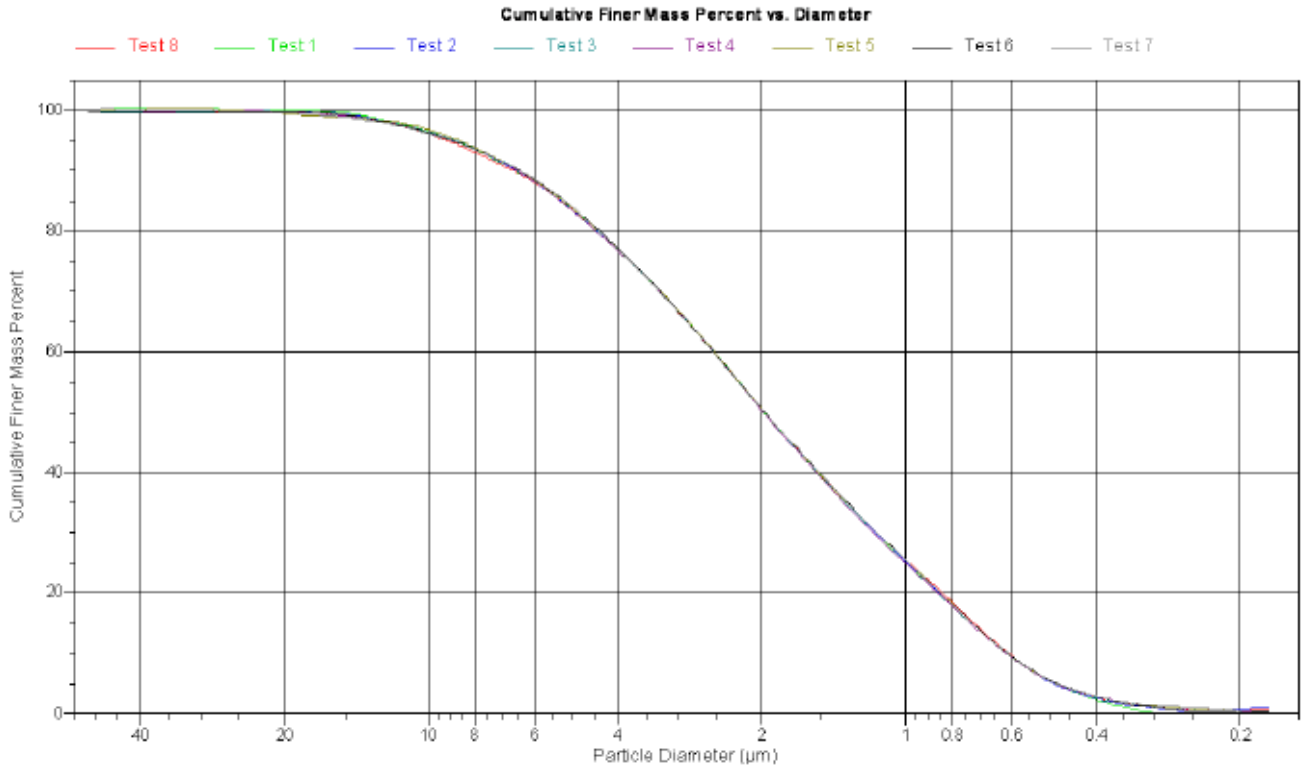
Particle Size Distribution of Talc



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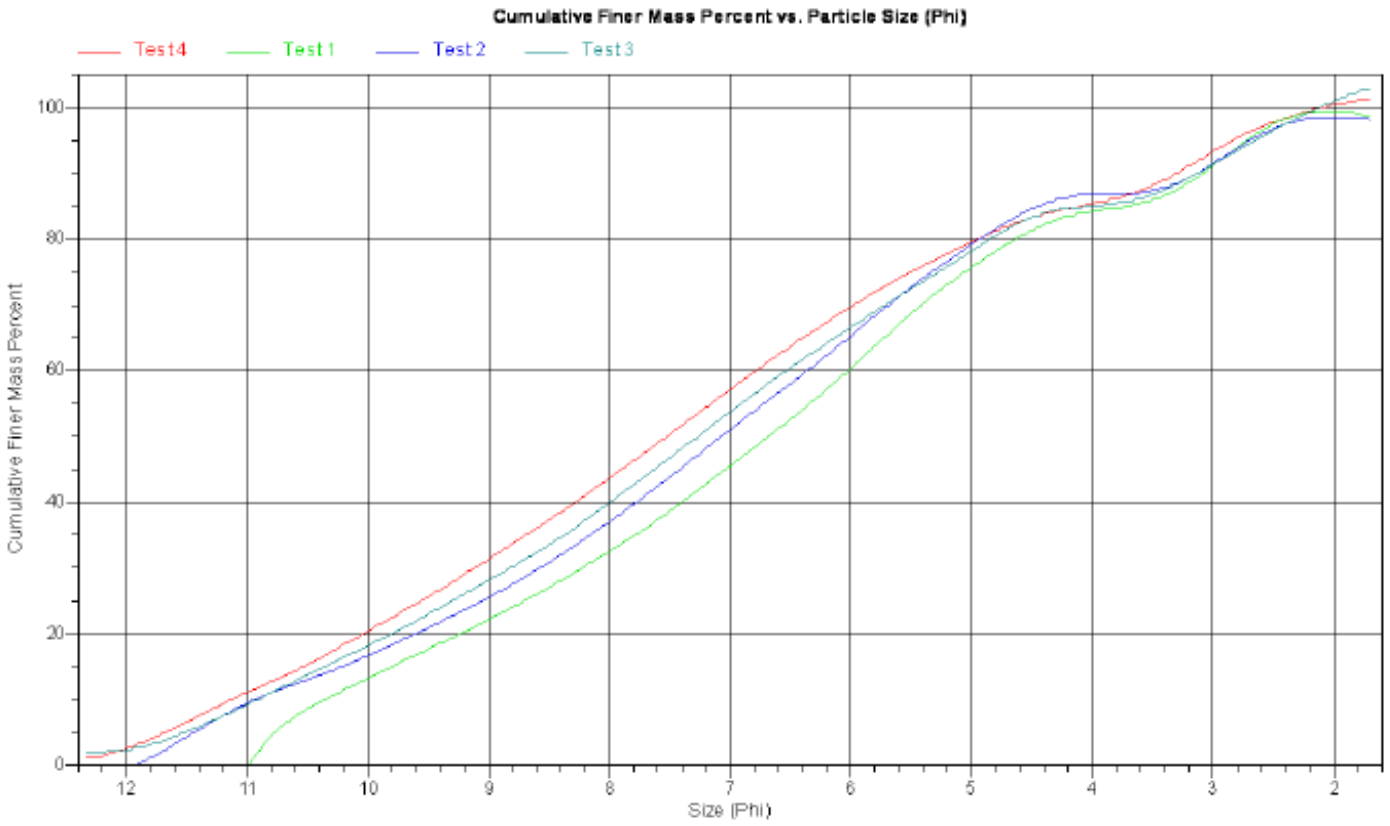
Particle Size Distribution of soil sample



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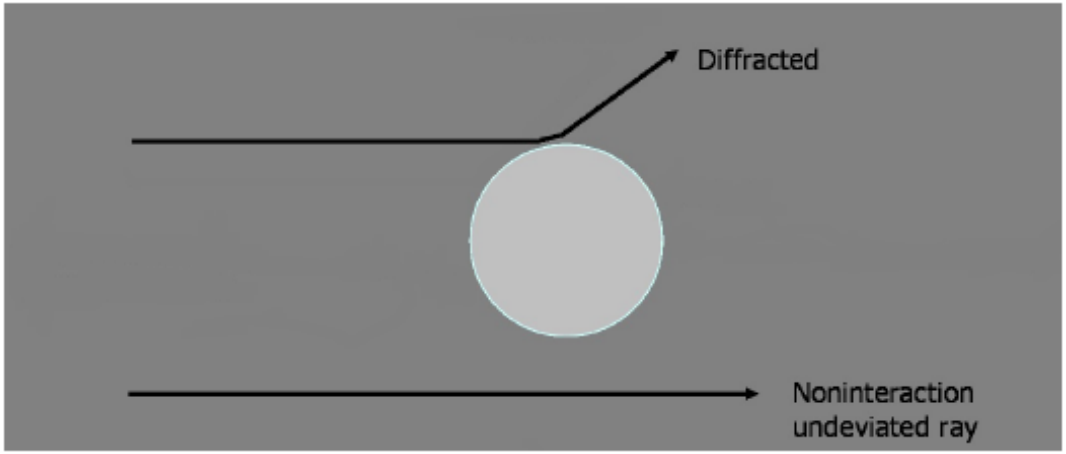
Static Laser Light Scattering – Saturn DigiSizer II 5205

Static Light Scattering

Light interaction with matter

- Diffraction – external surface of material only, Fraunhofer
- Refraction – light passing through material, changing direction
- Reflection – light interaction at phase boundaries, internal and external, changing directions
- Absorption – light interacting with material, with loss of light energy



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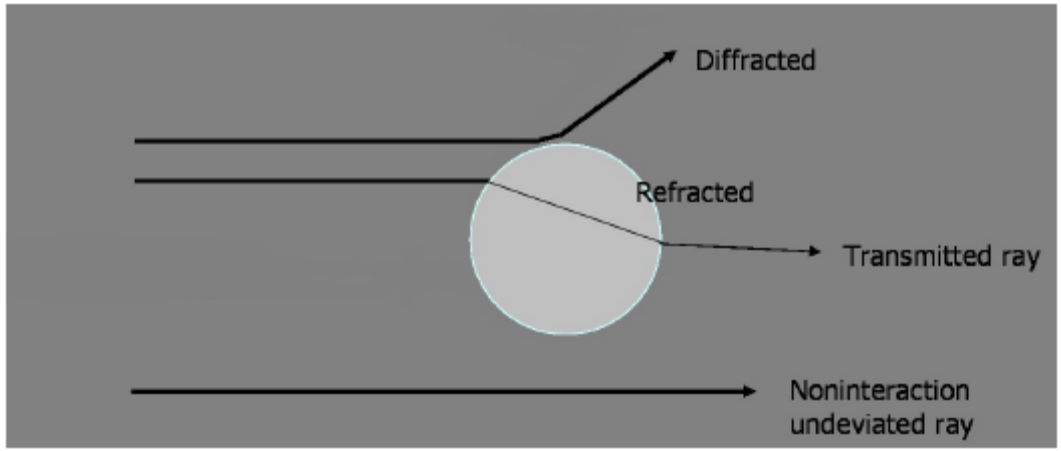

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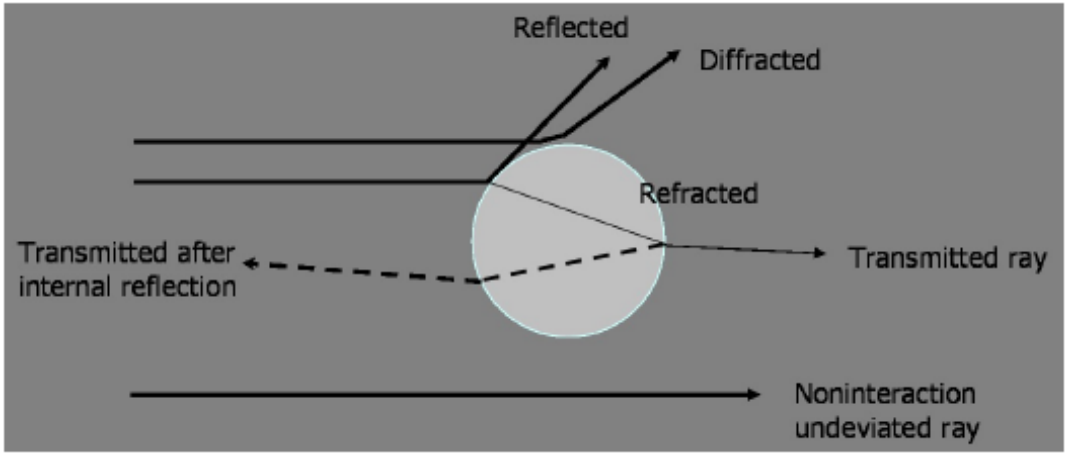

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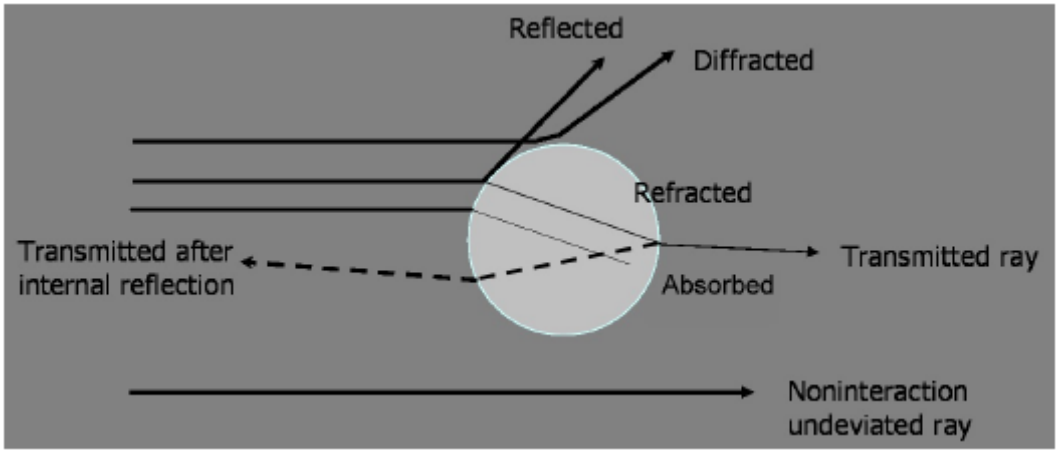
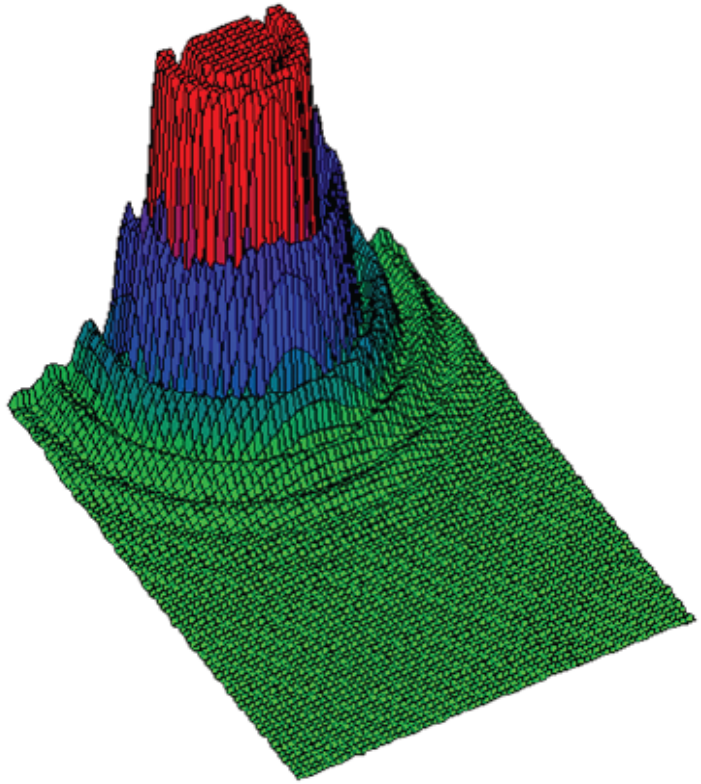


Particle Size Distribution Analysis

Static Light Scattering

- Particles scatter light in all directions
- Intensity pattern depends on particle size
- Assumes spherical particle shape
- Widely used fast technique that can be applied to various particulate systems
- Easily automated in a variety of commercially available instruments



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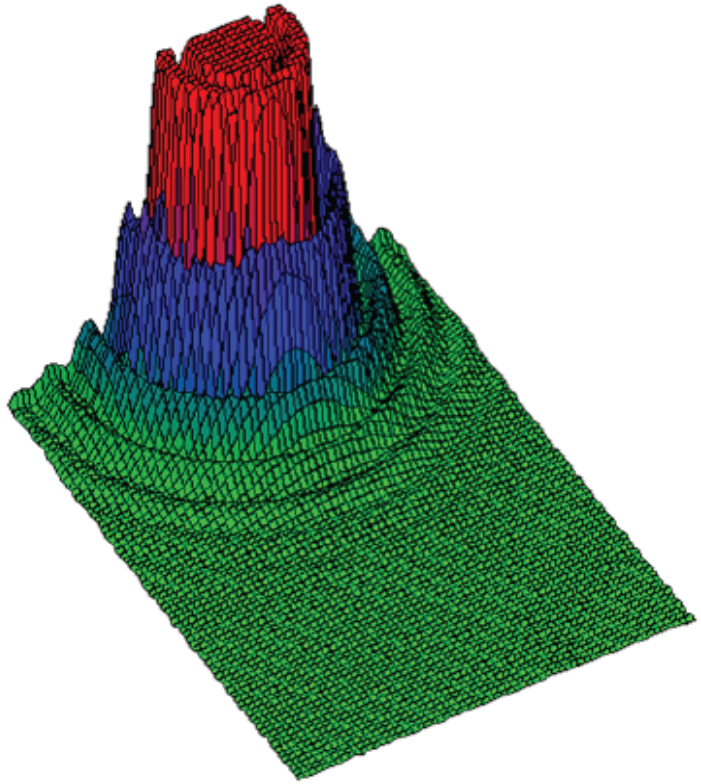

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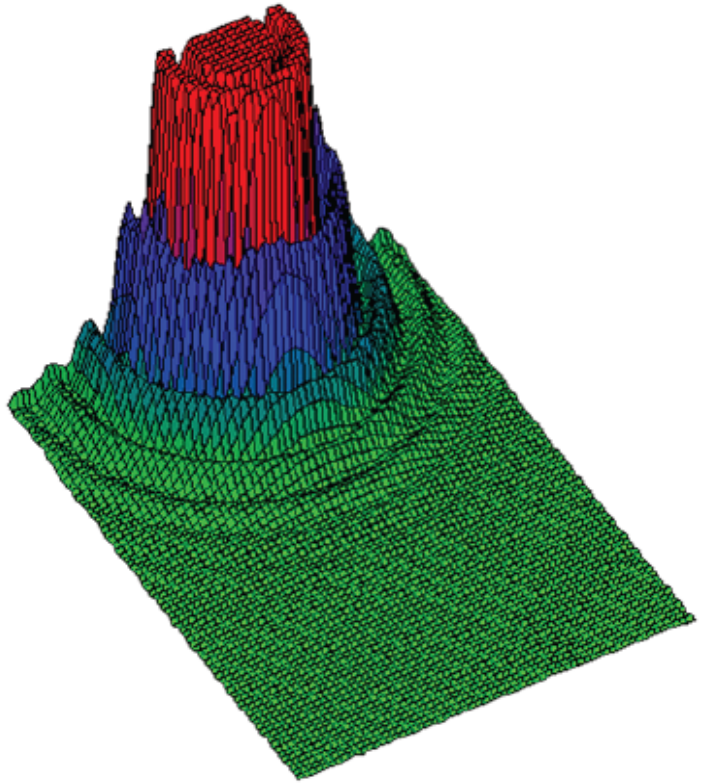

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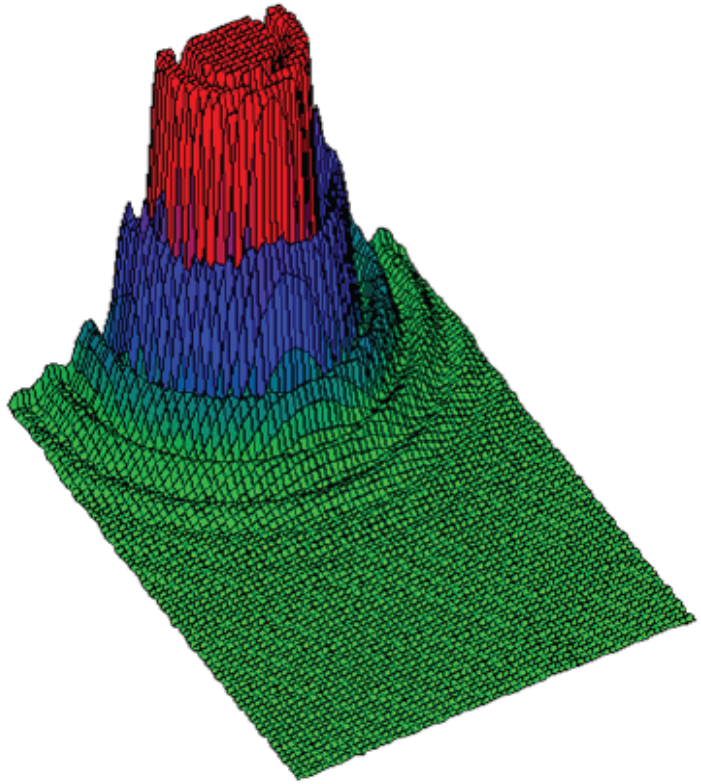

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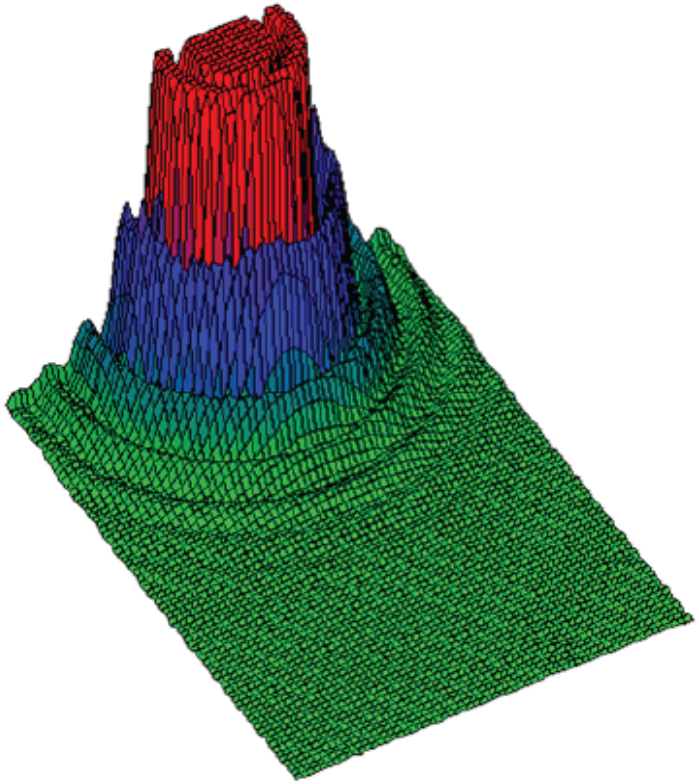
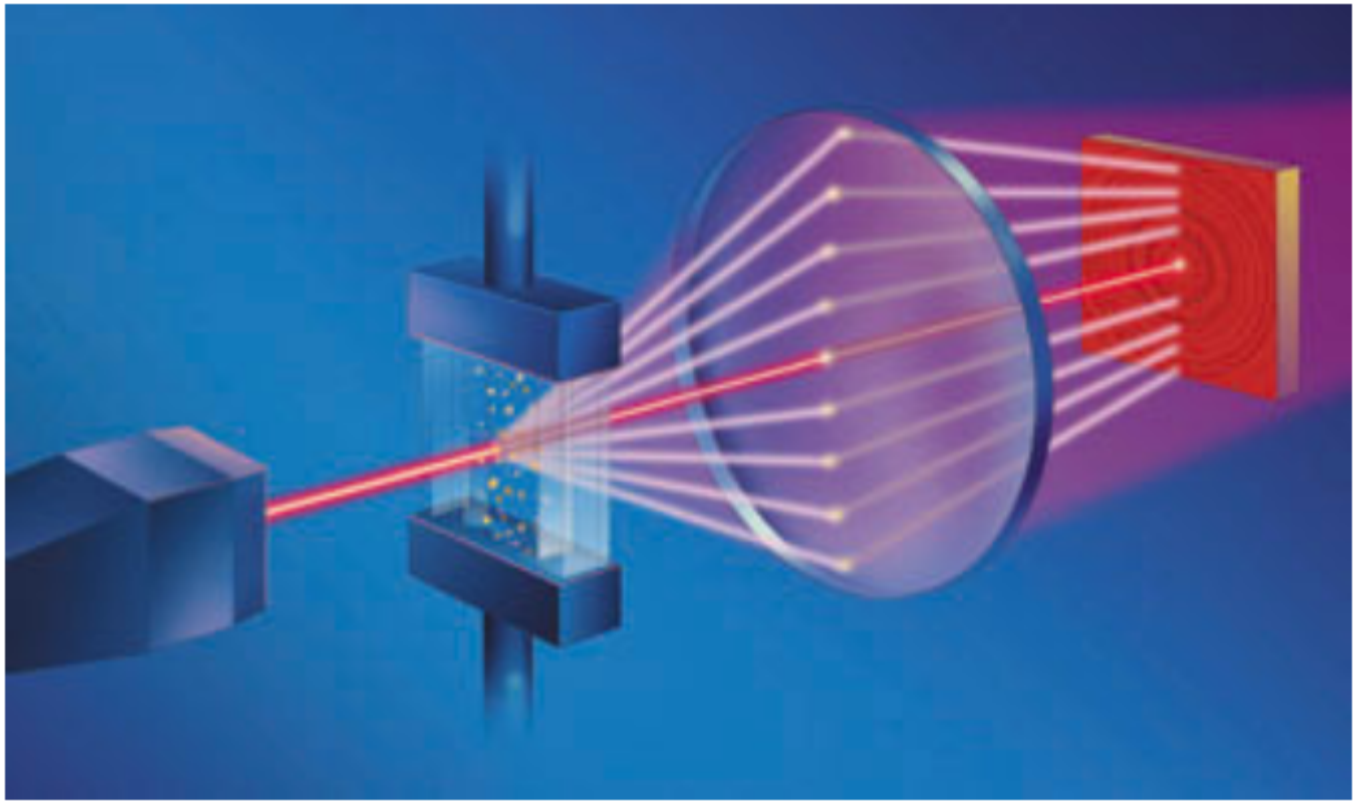
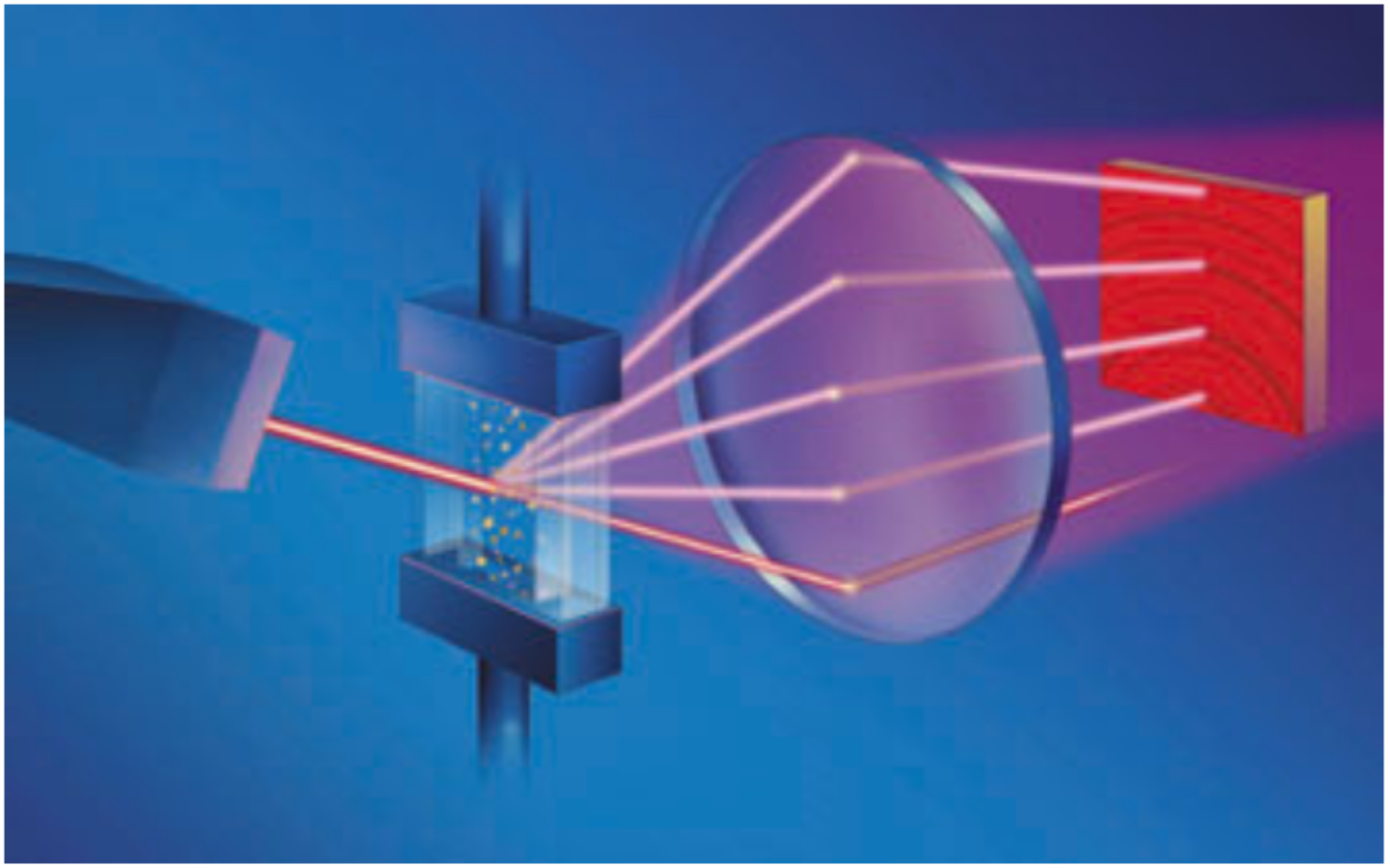


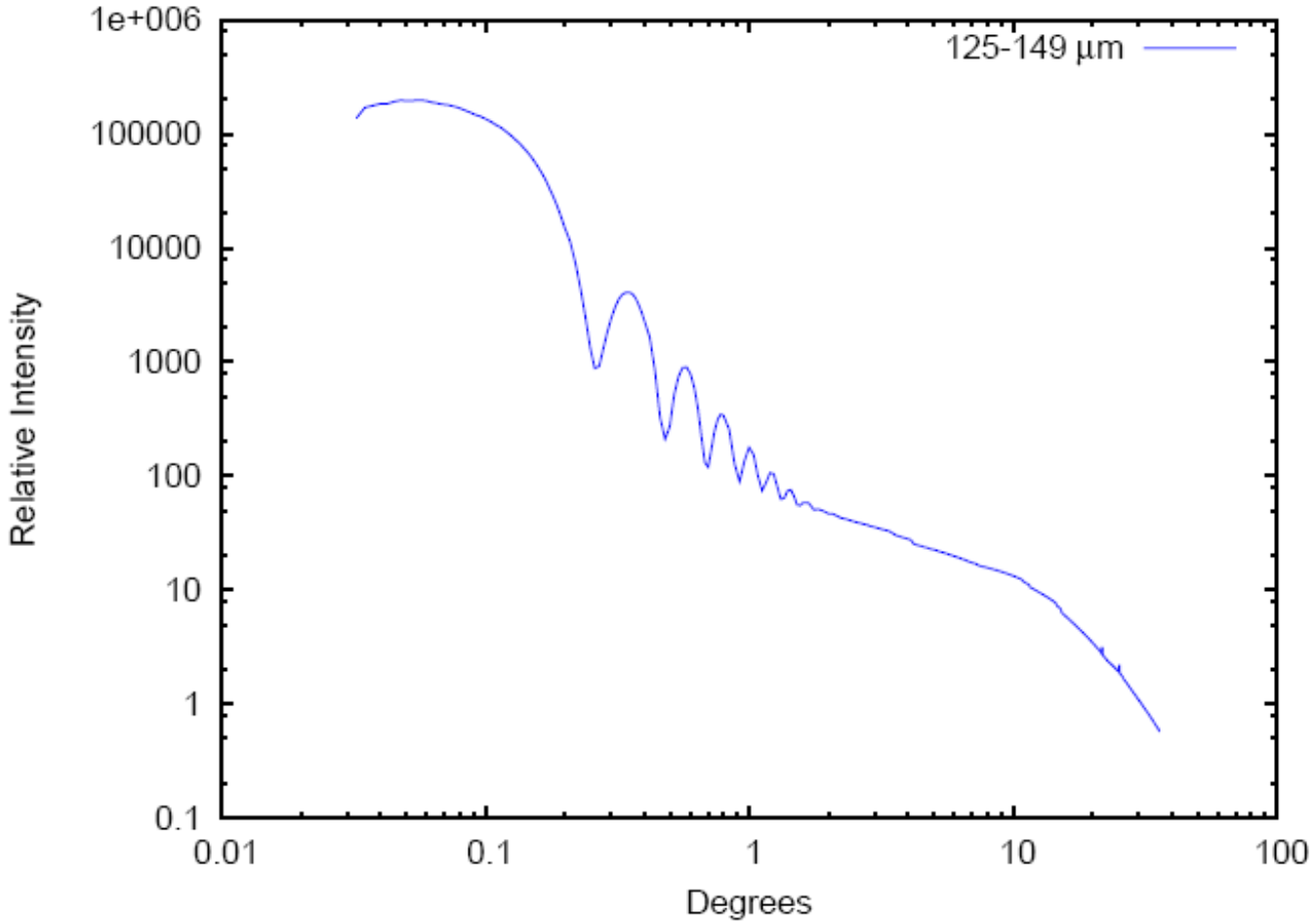
Fraunhofer Diffraction, 1840



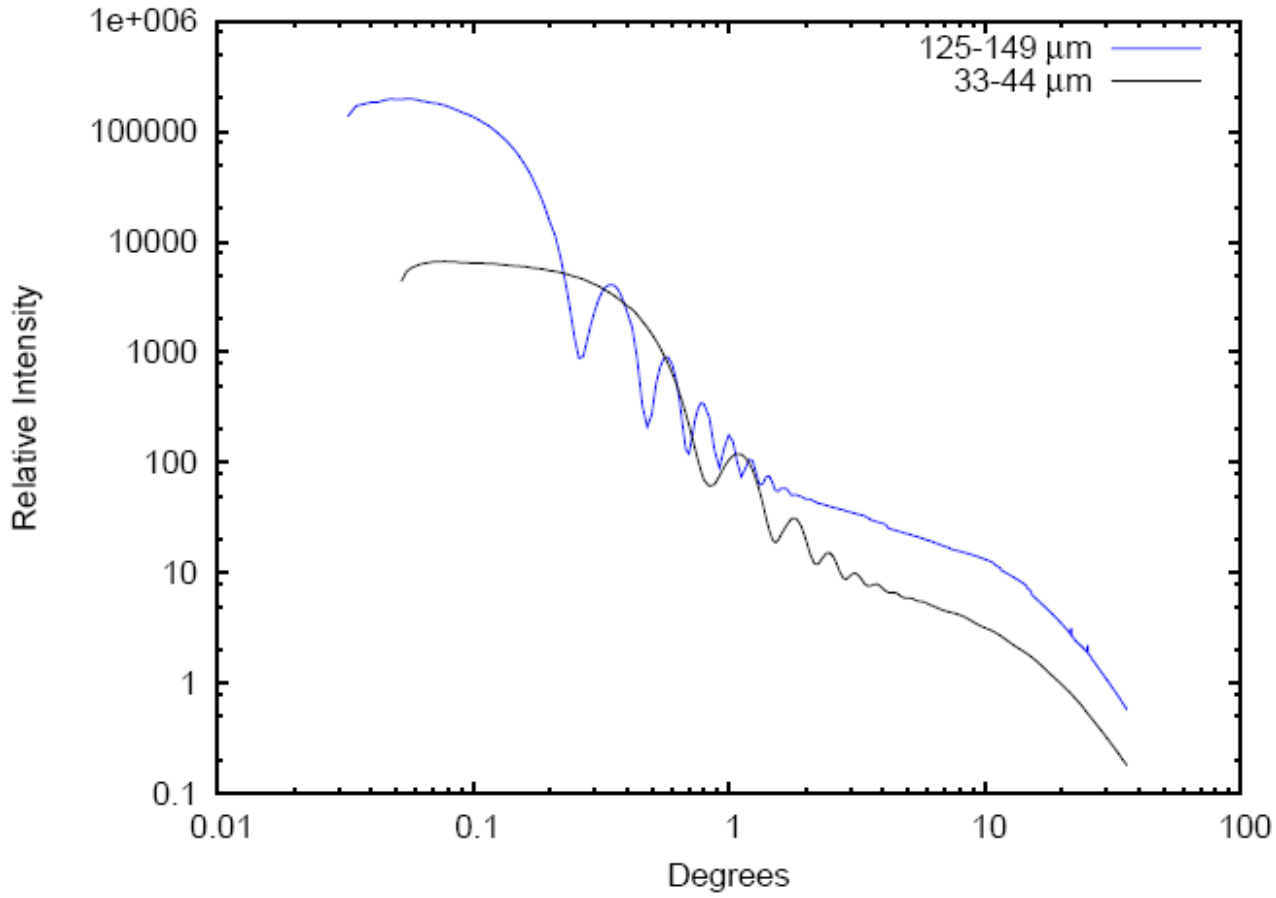
Mie Scattering, 1906



Scattering patterns – large spheres



Scattering patterns – large and medium spheres



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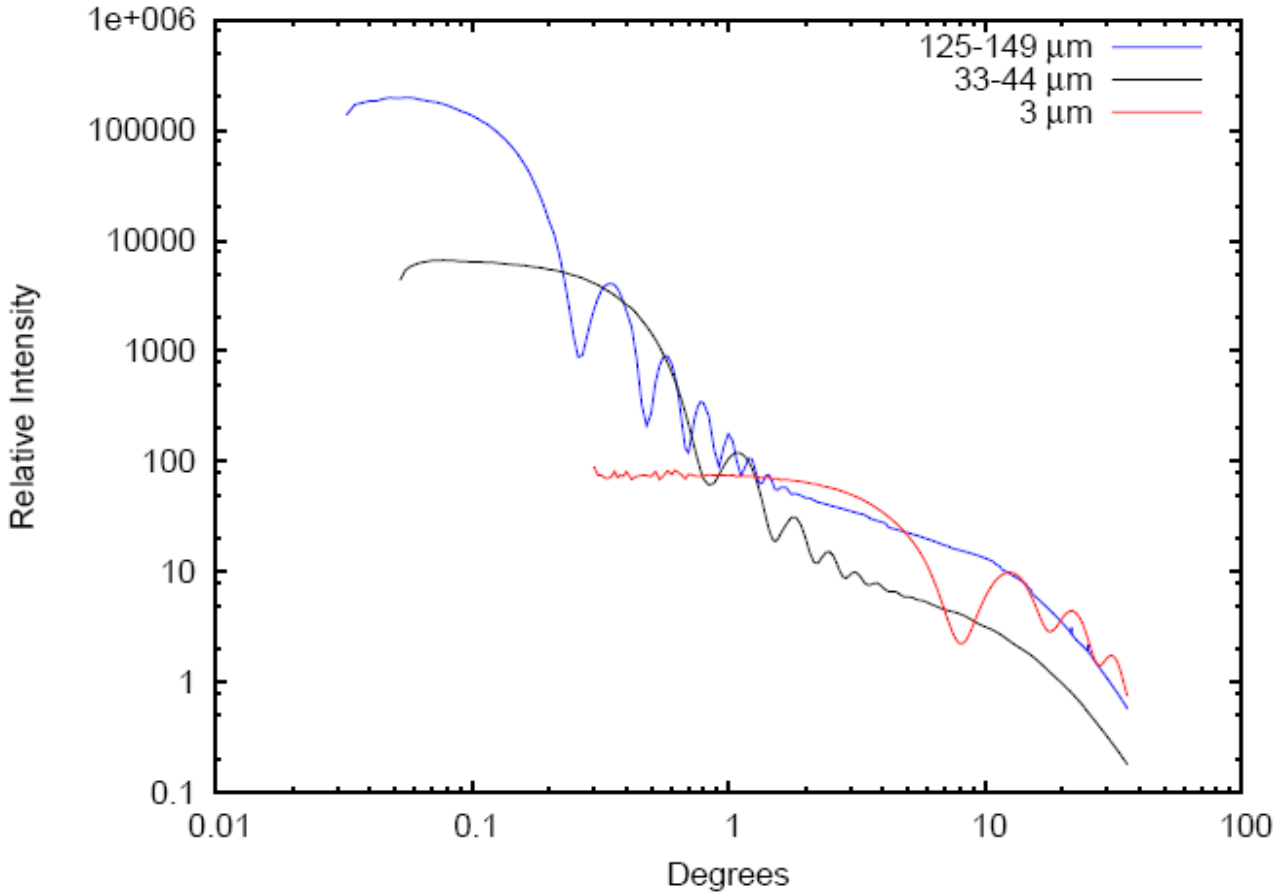
Scattering patterns – large, medium, and small spheres



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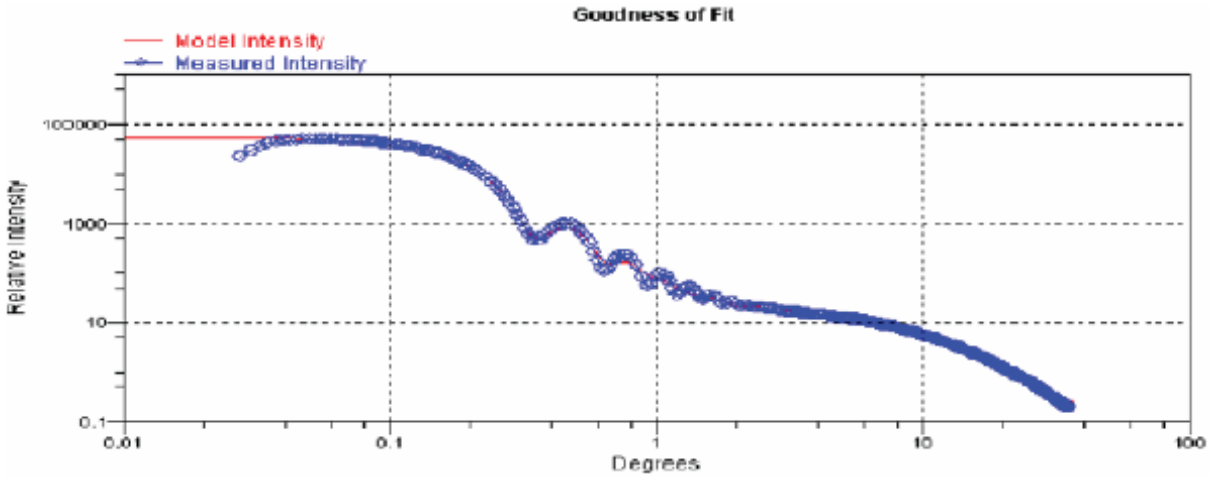
Particle Size Distribution Analysis

Laser Light Scattering

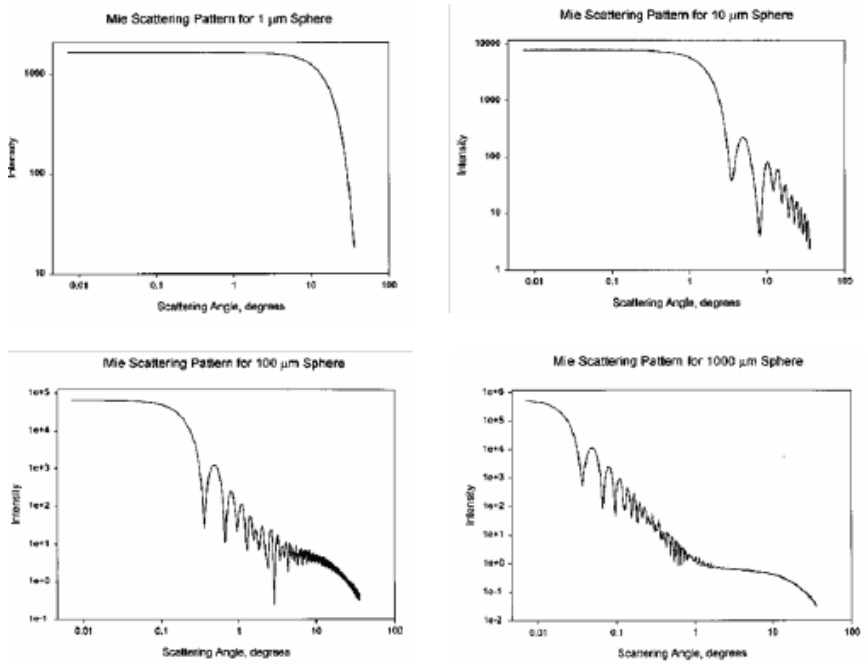
- Groups of particles scatter light essentially equal to the sum of light scattered by the individual particles.
- Particle size distribution calculated using mathematical deconvolution with optical models for selected particle sizes.
- Remember that agglomerates and aggregates look like large particles.



Static Laser Light Scattering

Optical Scattering Models

- Group of specific scattering patterns
- One model for each particle size class
- For a given relative complex reflective index
- Used to calculate particle size distribution



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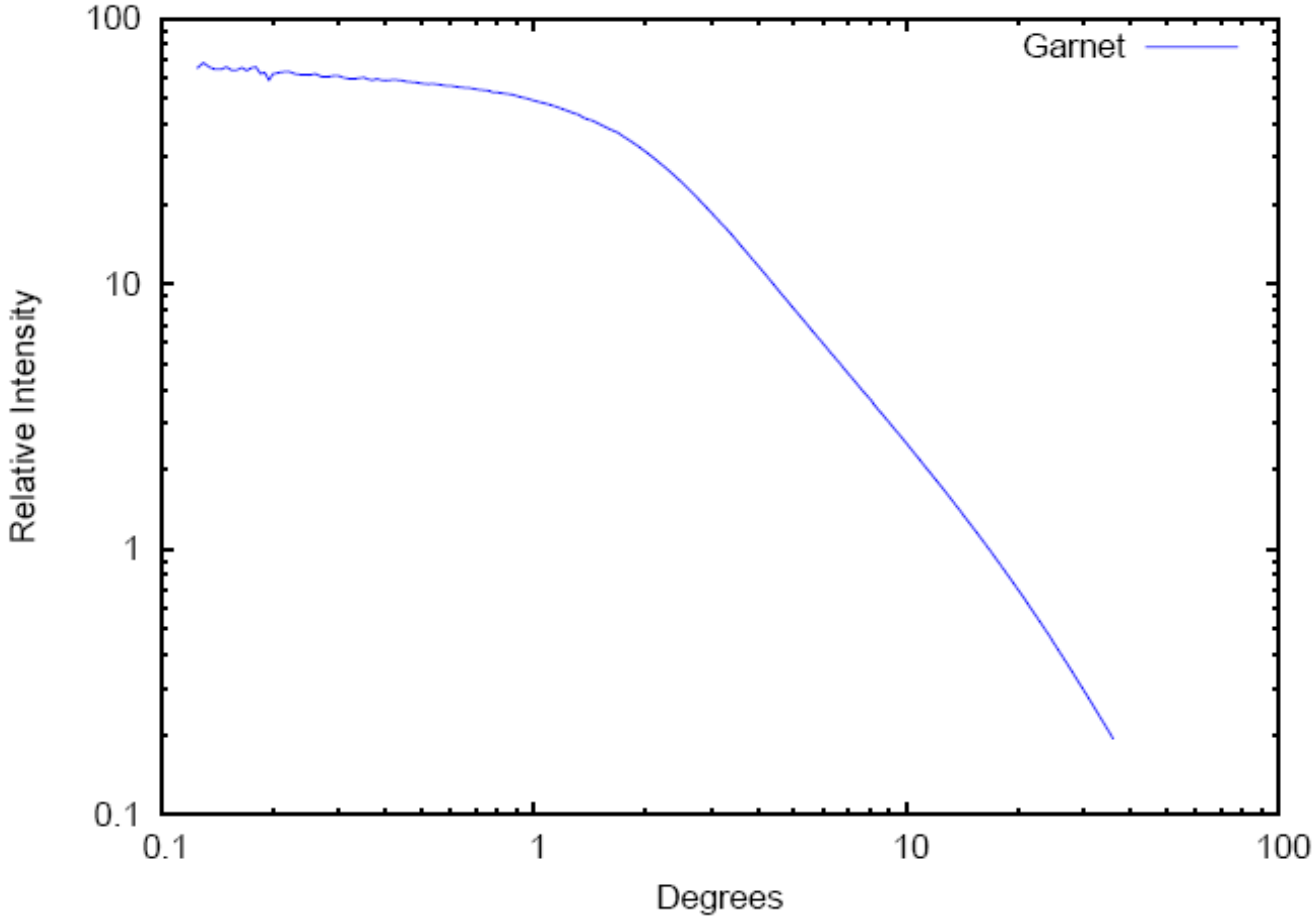
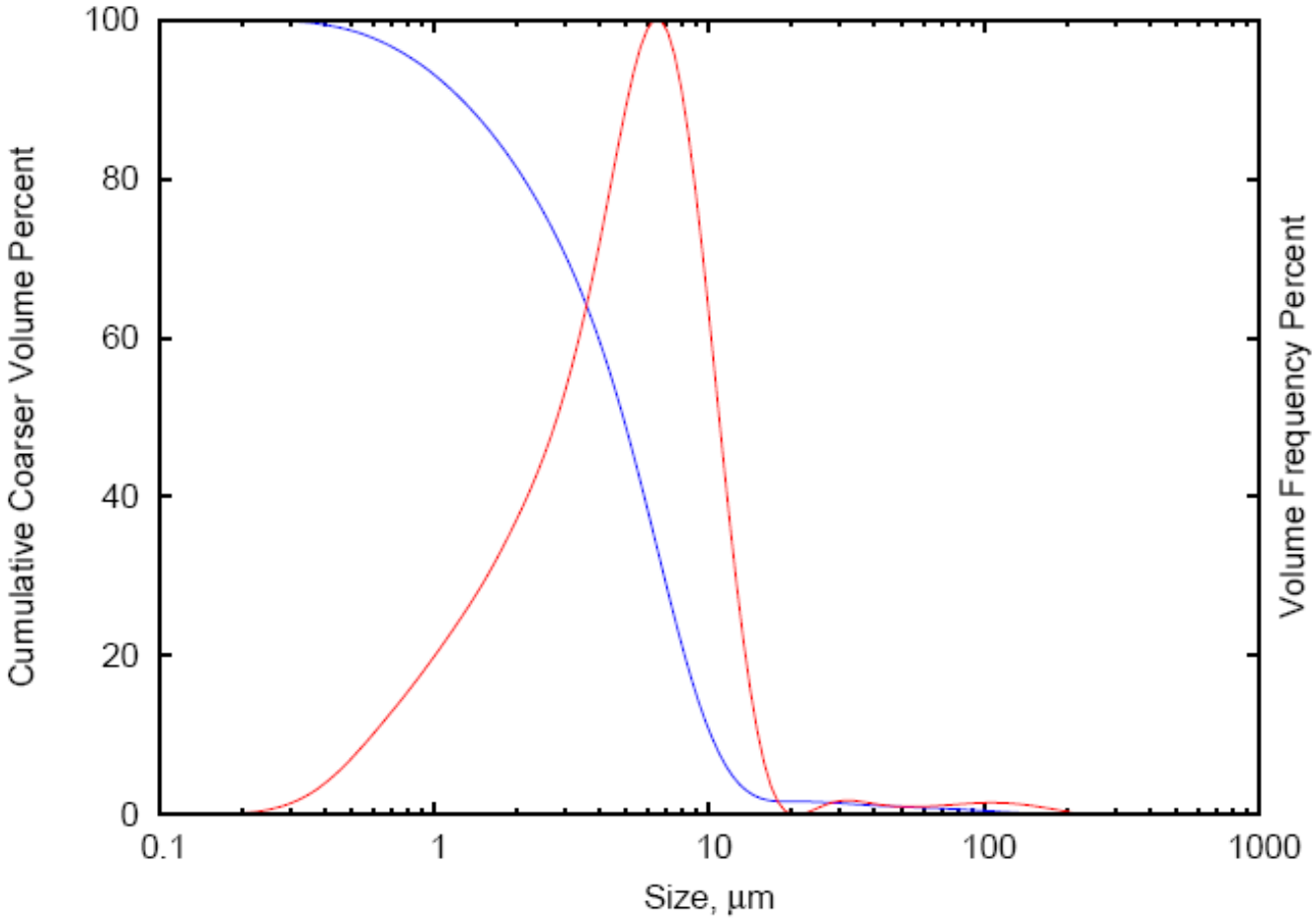
Garnet scattering pattern



Garnet particle size distribution



Static Laser Light Scattering

Quality of Results

Resolution → number of detector elements

Static Laser Light Scattering

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Resolution	→	number of detector elements
Sensitivity	→	linearity of detector elements

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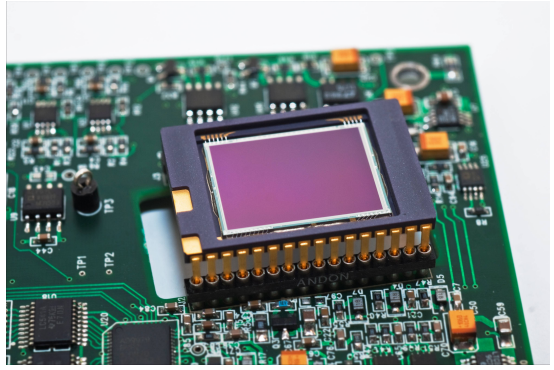
Charge-coupled Device

- Over 8.3 million detector elements
- Five degrees of scattering pattern
- Automatically aligning



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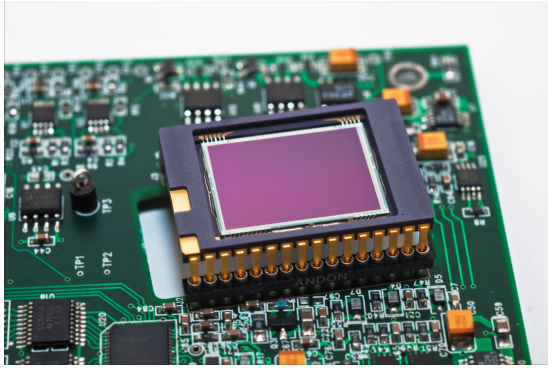

Static Laser Light Scattering

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Charge-coupled Device

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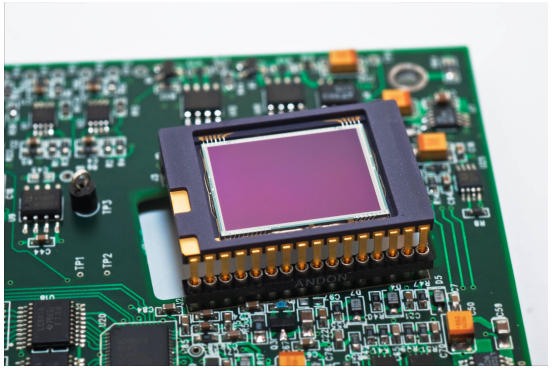
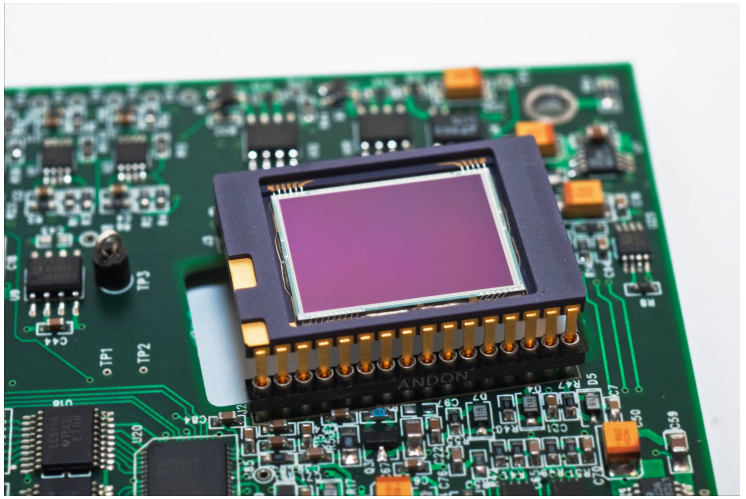
- Fourteen incident laser positions



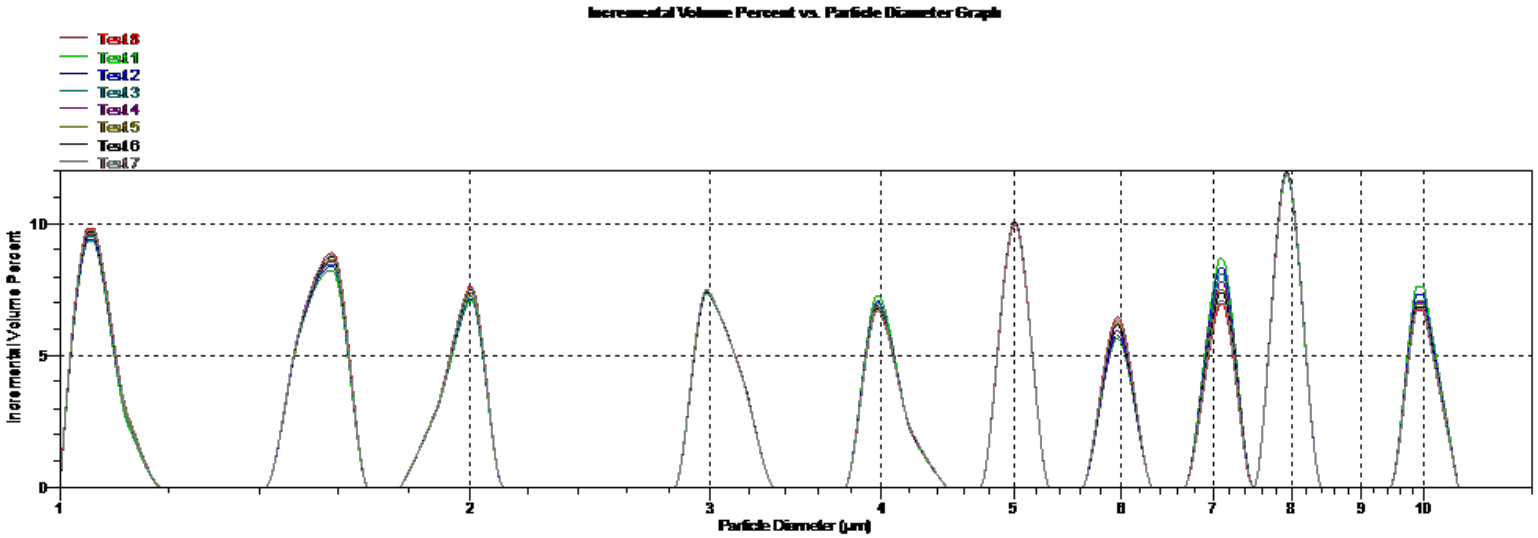
Static Laser Light Scattering

Optical Scattering Models

- Group of specific scattering patterns – 592 angle classes from 0.0025 – 48 degrees
- One model for each particle size class – 193 size classes from 0.040 – 2500 µm
- For a given relative complex reflective index
- Used to calculate particle size distribution



Particle Size Distribution Resolution



Mix of 10 Monodisperse Polystyrene Standards

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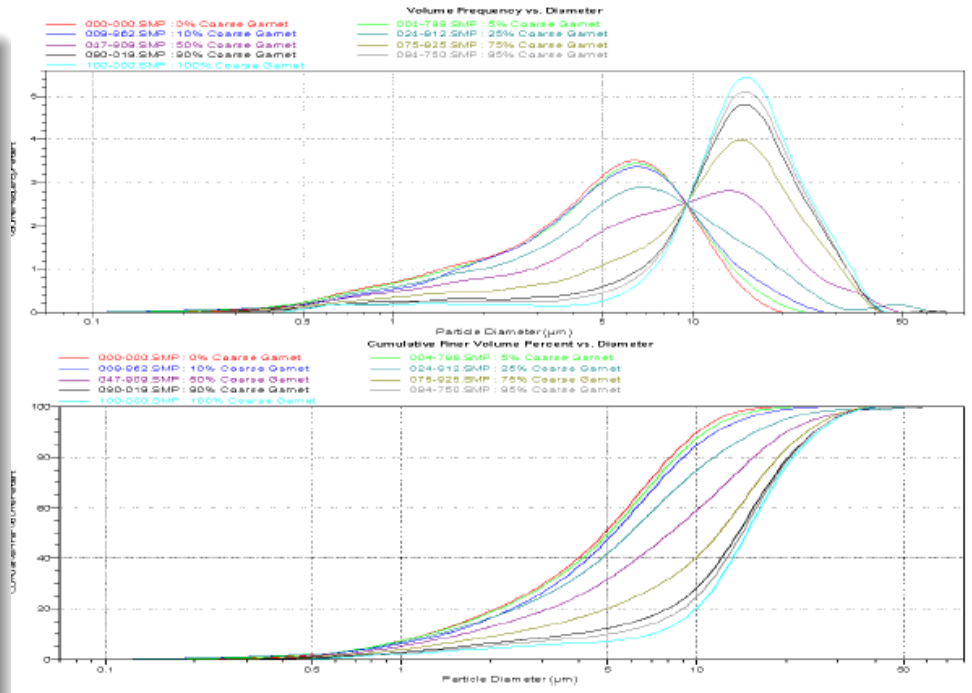
Particle Size Distribution Sensitivity

Blends of Two Samples

- Medium garnet – 6.330 μm mode
- Coarse garnet – 15.00 μm mode
- Demonstrates ability to see small changes in amount of material at each diameter



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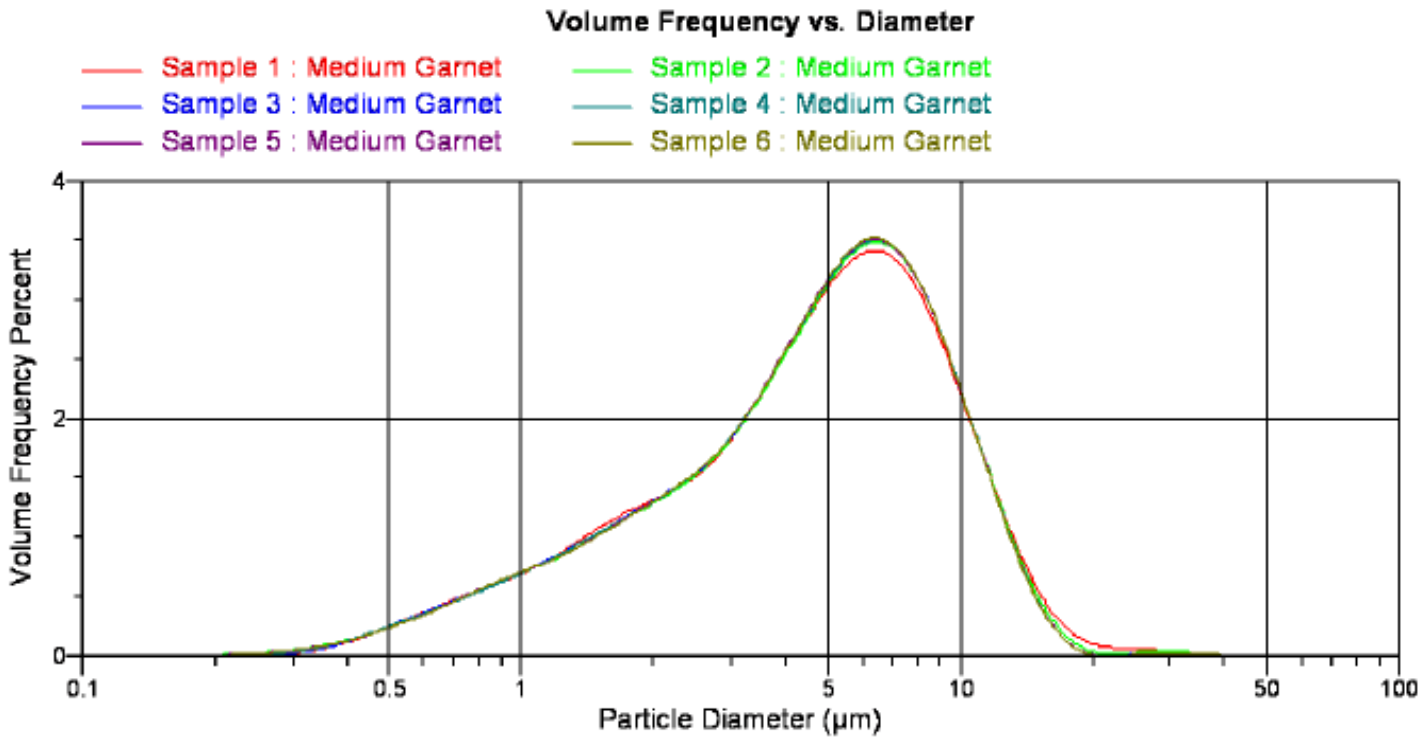
Particle Size Distribution Repeatability



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Particle Size Distribution Reproducibility

Reproducibility Study

- Traceable Garnet Reference Material
- Over 30 instruments, on different continents
- Multiple analyses from each instrument

Garnet Specifications

Statistic	Central Value	Tolerance
Mean Diameter	5.30 μm	0.12 μm
Median Diameter	4.85 μm	0.10 μm
90th Percentile	9.94 μm	0.30 μm
10th Percentile	1.210 μm	0.082 μm

Particle Size Distribution Reproducibility

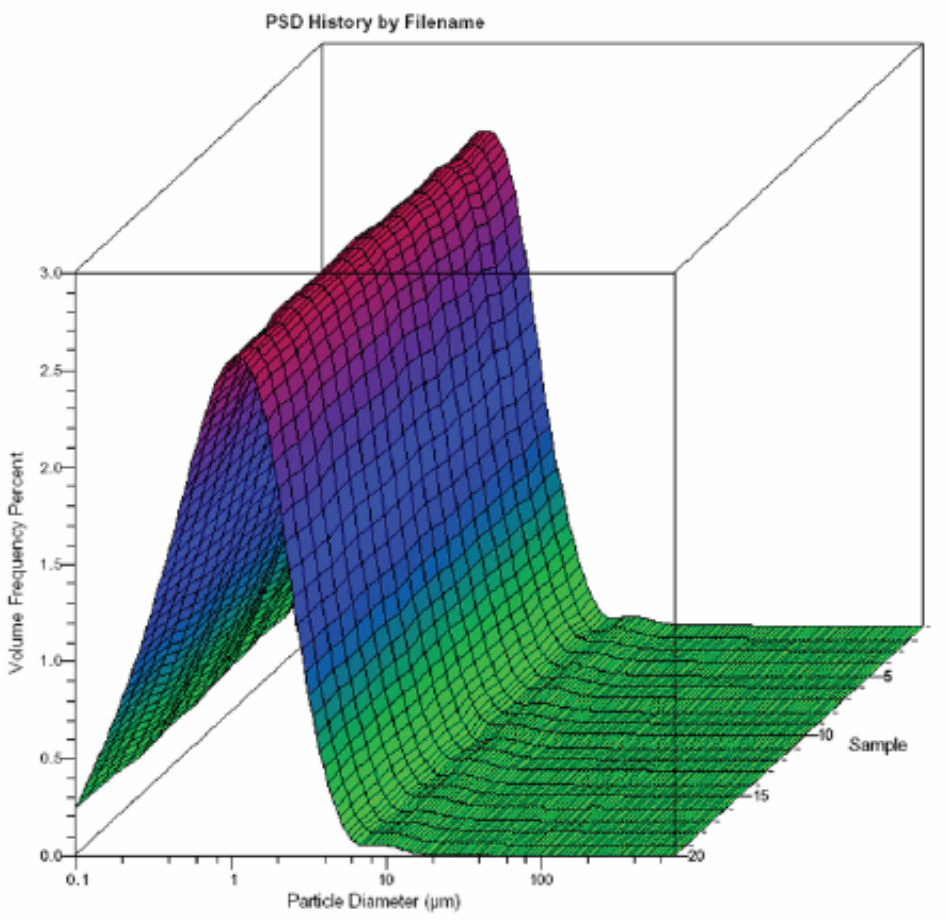
Results from 32 Instruments

Statistic	Central Value	Tolerance
Mean Diameter	5.299 μm	0.035 μm
Median Diameter	4.861 μm	0.033 μm
90th Percentile	9.947 μm	0.072 μm
10th Percentile	1.254 μm	0.015 μm

Garnet Specifications

Statistic	Central Value	Tolerance
Mean Diameter	5.30 μm	0.12 μm
Median Diameter	4.85 μm	0.10 μm
90th Percentile	9.94 μm	0.30 μm
10th Percentile	1.210 μm	0.082 μm

Overlay of Sample Analysis of 20 Instruments



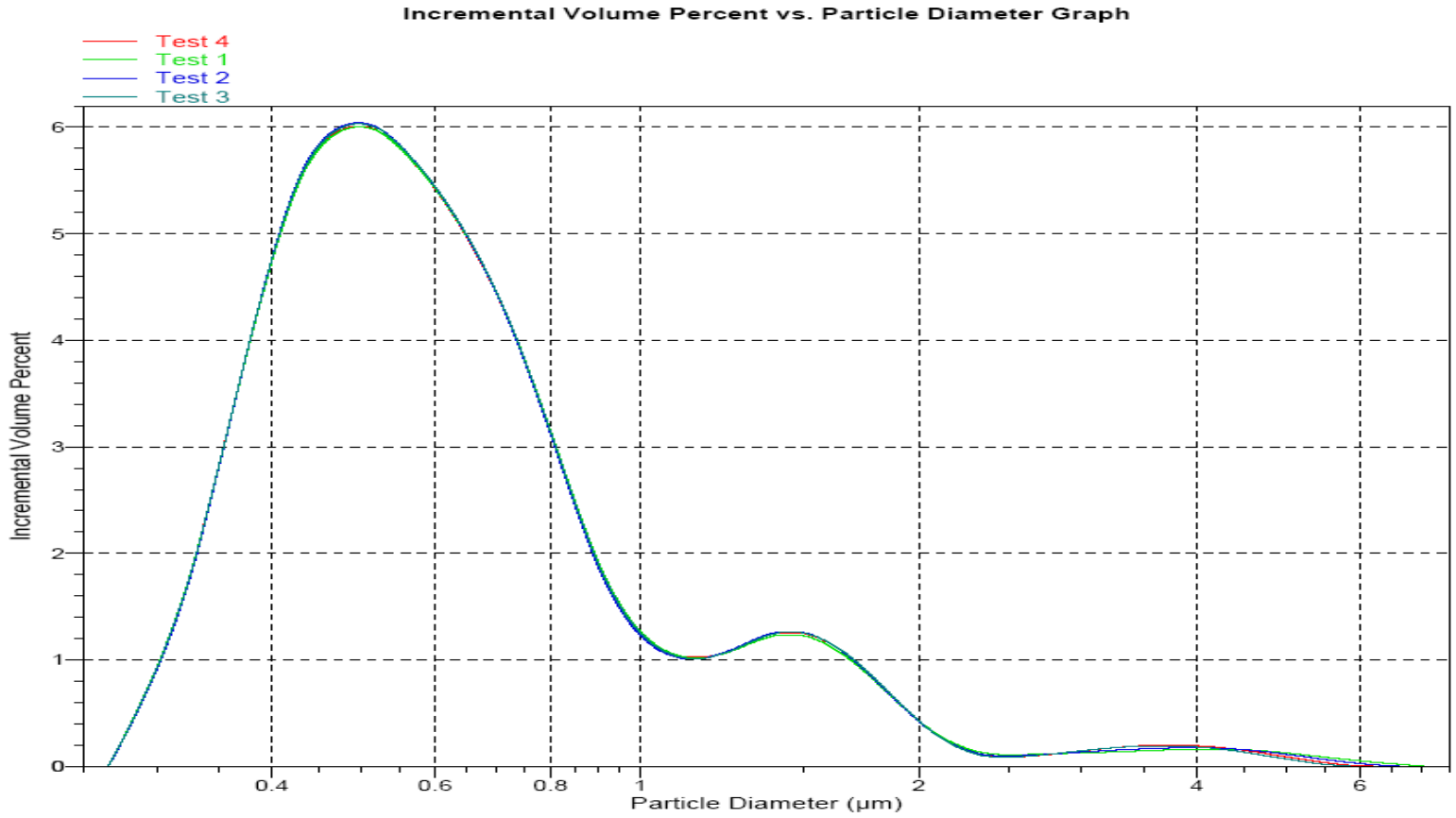
materials characterization WORKSHOP

Static Laser Light Scattering

Applications

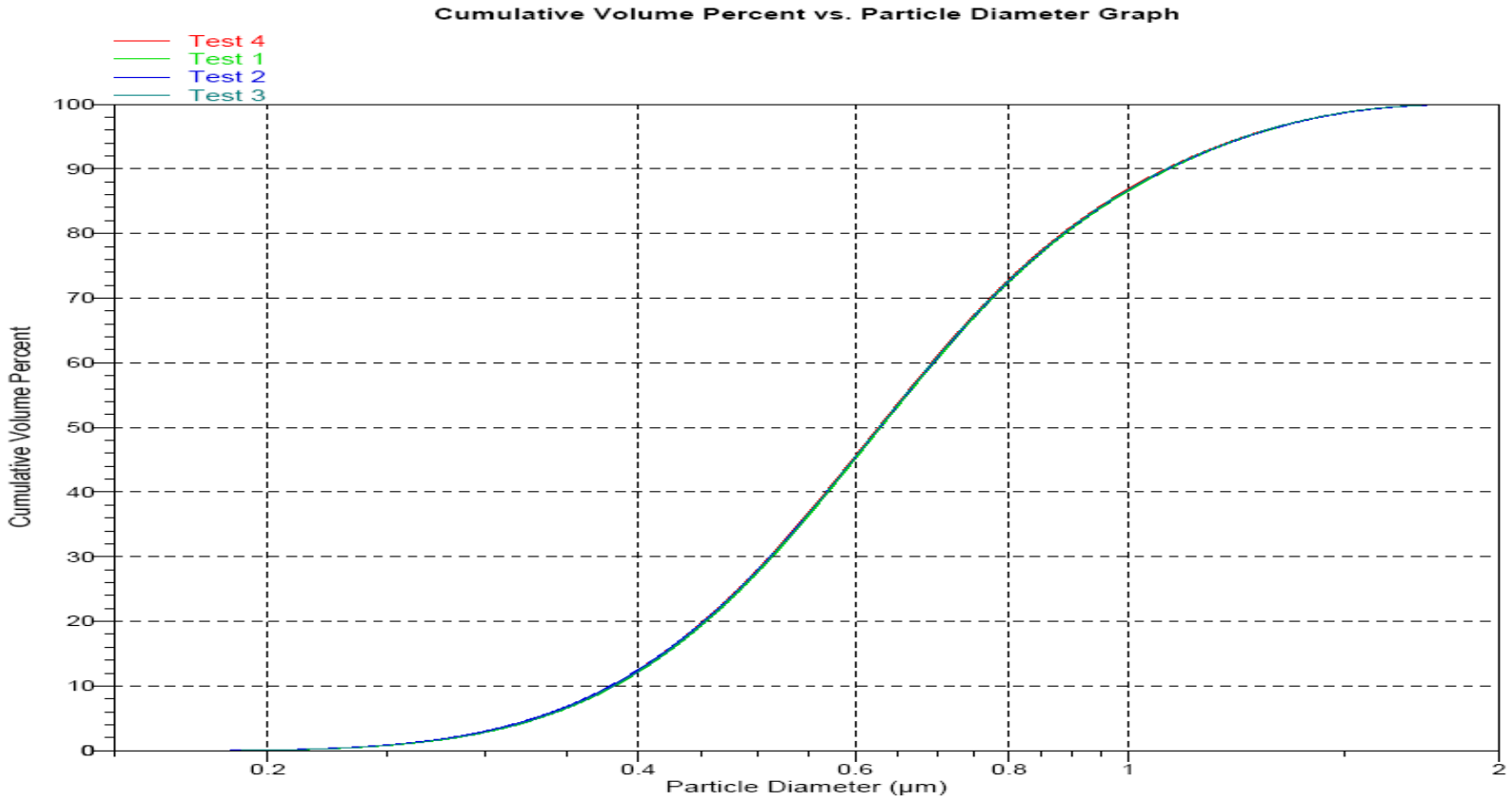
- Samples with narrow distributions where size resolution is needed.
- Samples where sensitivity to amount at each size is critical.
- Samples with broad distributions where a wide analysis range are needed.
- Production environments where fast analyses are needed.
- Multiple locations where reproducibility is needed.

Saturn DigiSizer II 5205



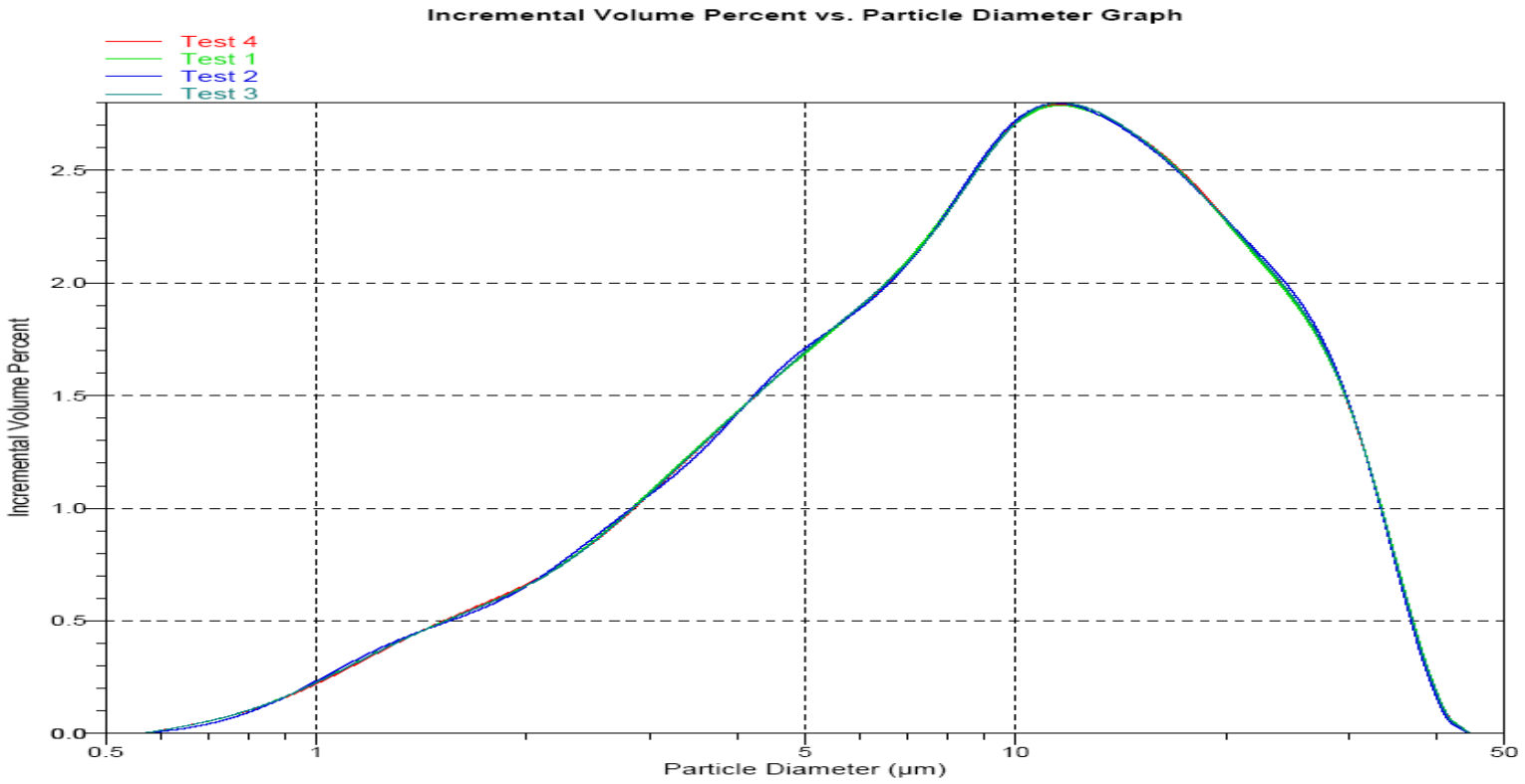
- Titanium Dioxide – Incremental Volume Percent

Saturn DigiSizer II 5205



- Zinc Oxide – cumulative volume percent

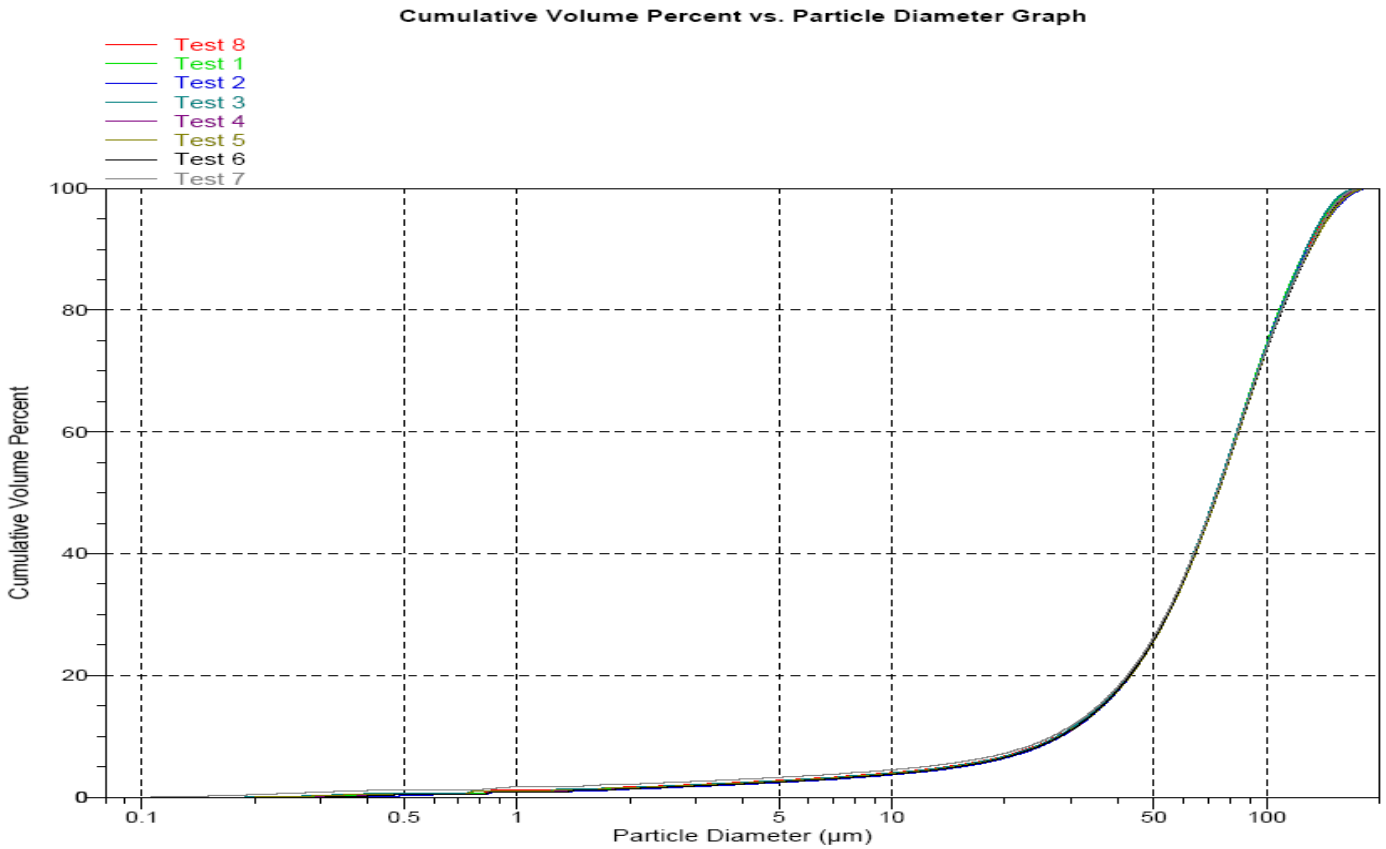
Saturn DigiSizer II 5205



- Magnesium Stearate

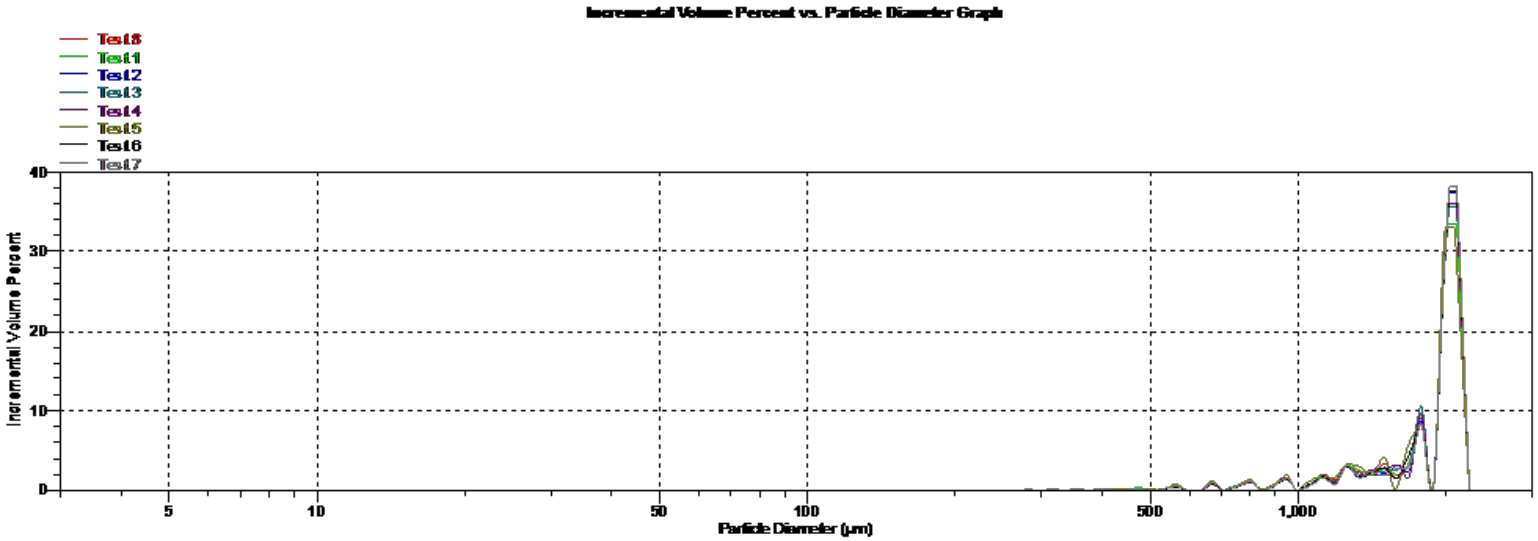
materials characterization WORKSHOP

Saturn DigiSizer II 5205



- Acetaminophen

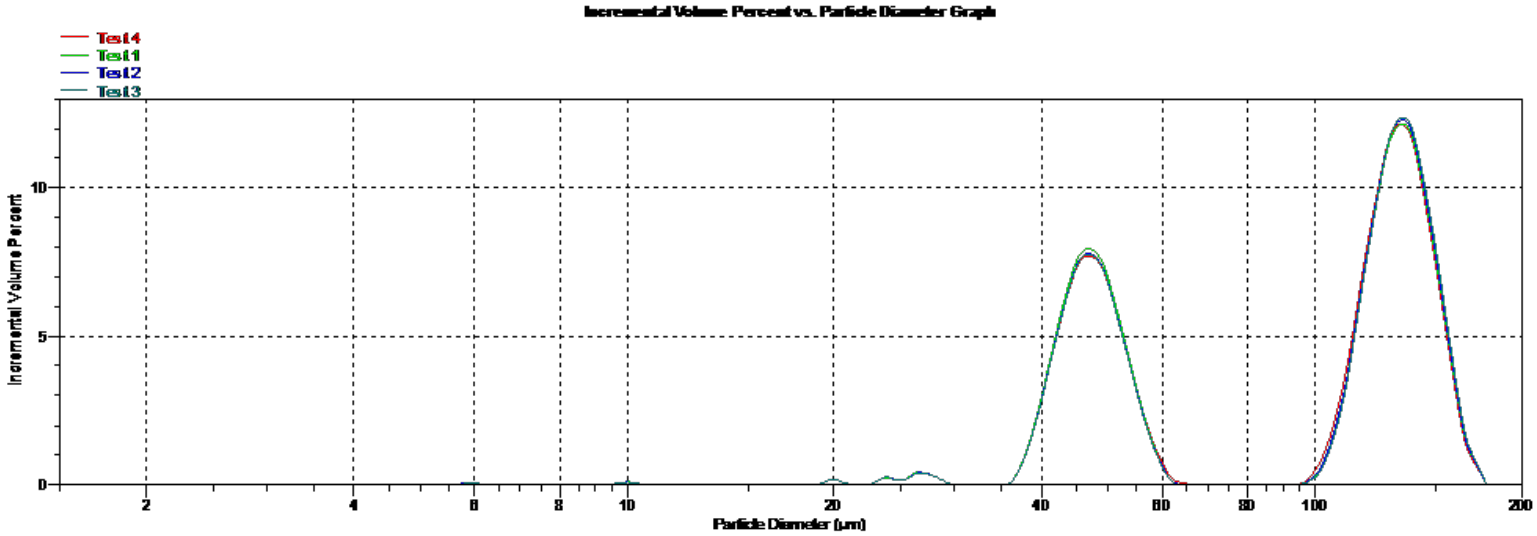
Saturn DigiSizer II 5205



- 2.1 mm Glass Beads

materials characterization WORKSHOP

Saturn DigiSizer II 5205



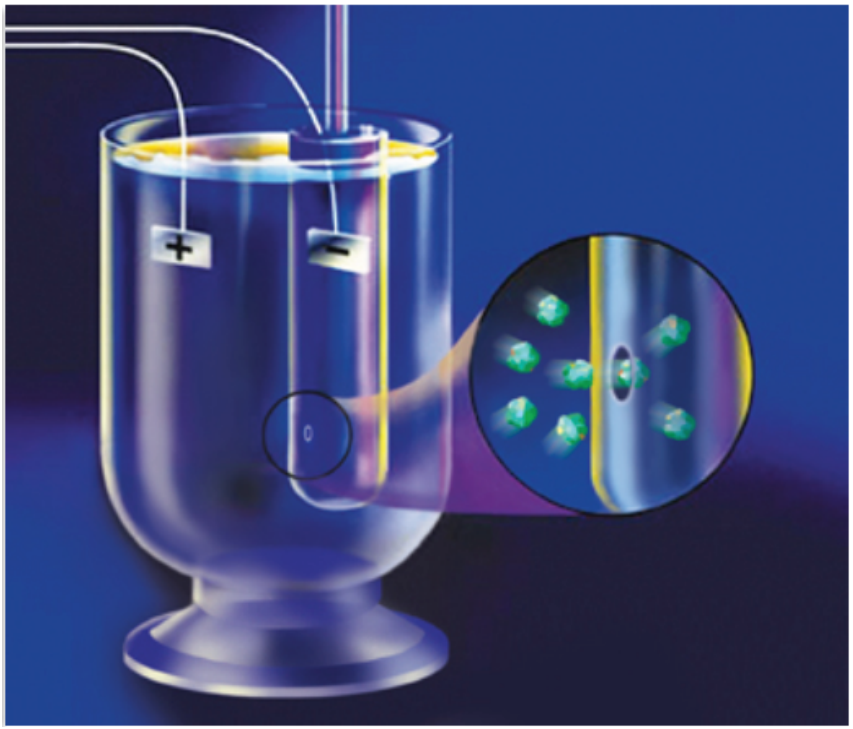
Glass Bead Blend 44 – 53 µm & 125 – 149 µm

Electrical Sensing Zone – Elzone II 5390

Electrical Sensing Zone

Particle Counter

- Particles suspended in a conductive liquid.
- Pass through a narrow orifice one at a time.
- Lowers conductivity through the orifice.
- Voltage increases to keep orifice current constant.
- Voltage change proportional to particle volume.



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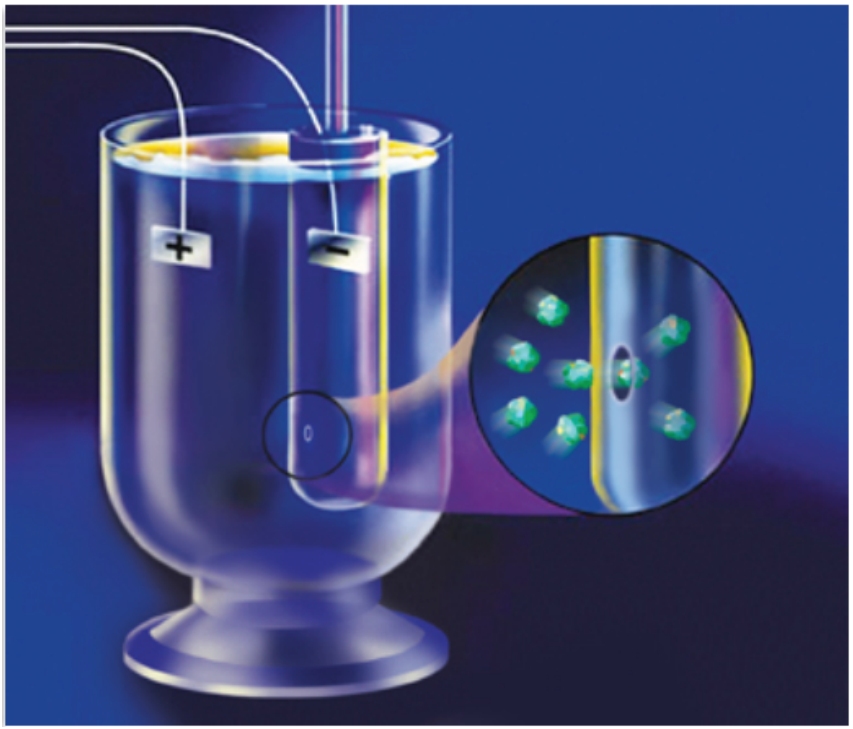

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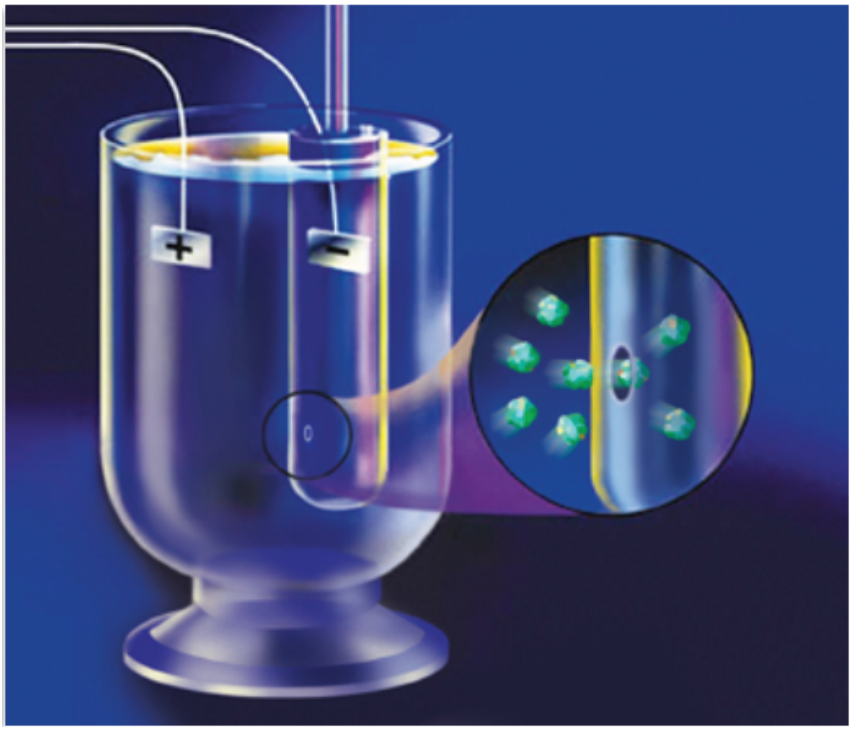

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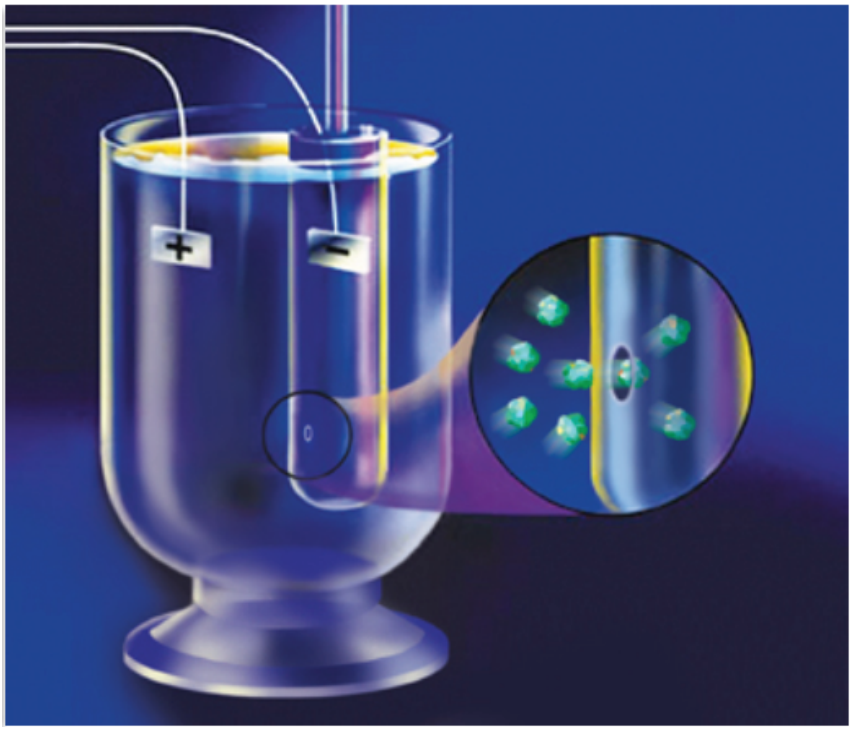

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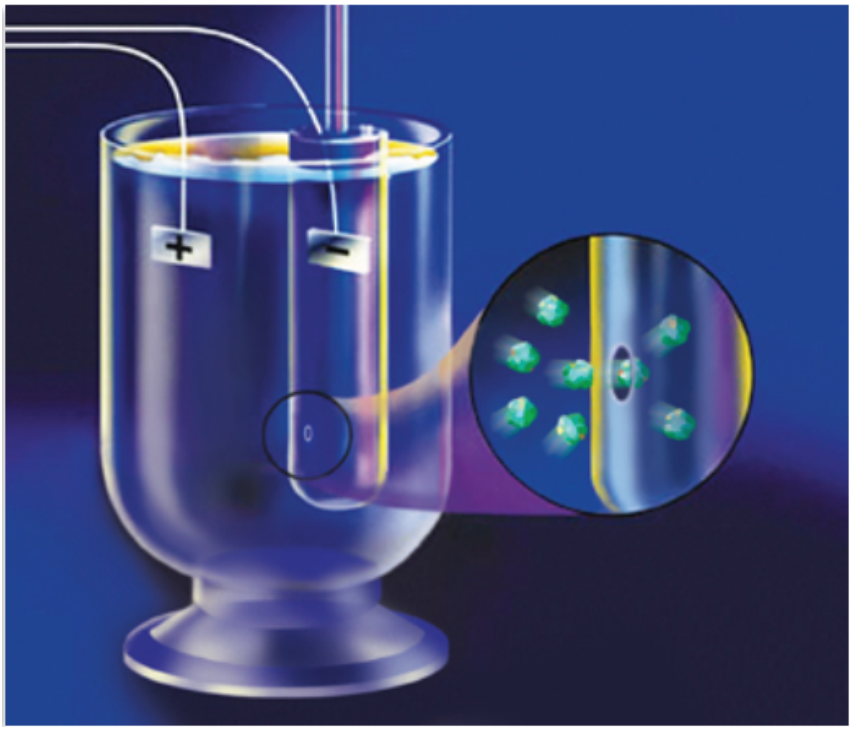


Electrical Sensing Zone

Advantages

- Counts and sizes organic and inorganic particles.
- Analyzes materials with mixed optical properties, densities and shapes.
- Higher resolution than most other sizing methods.
- Lower quantity of sample needed for accurate, easy analysis.
- Typical analysis time less than 2 minutes.

Electrical Sensing Zone

Analysis Modes

- Number, area, volume, or mass based distributions.
- Frequency or cumulative distributions.
- Absolute, relative, or concentration basis.
- Mass balance to account for fines or porosity.
- Pulse width versus particle size related to length of particles.

Electrical Sensing Zone

Analysis Modes

- Number, area, volume, or mass based distributions.
- Frequency or cumulative distributions.
- Absolute, relative, or concentration basis.
- Mass balance to account for fines or porosity.
- Pulse width versus particle size related to length of particles.

Data Presentation

- Merge data from multiple orifice tubes into single distribution.
- Add multiple analyses to increase total particle count.
- Average multiple analyses to increase statistical reliability.

Electrical Sensing Zone

Applications

Bio-cells	Emulsions	Abrasives
Pigments	Toners	Inks
Filtration products	Separation media	Concentration analyses

Ideal for:

- Whenever counting, small quantity, or mixed or unknown properties limit other techniques.
- Whenever presence of a few outliers, especially at large diameters, must be determined.

Elzone II 5390 Pisces option

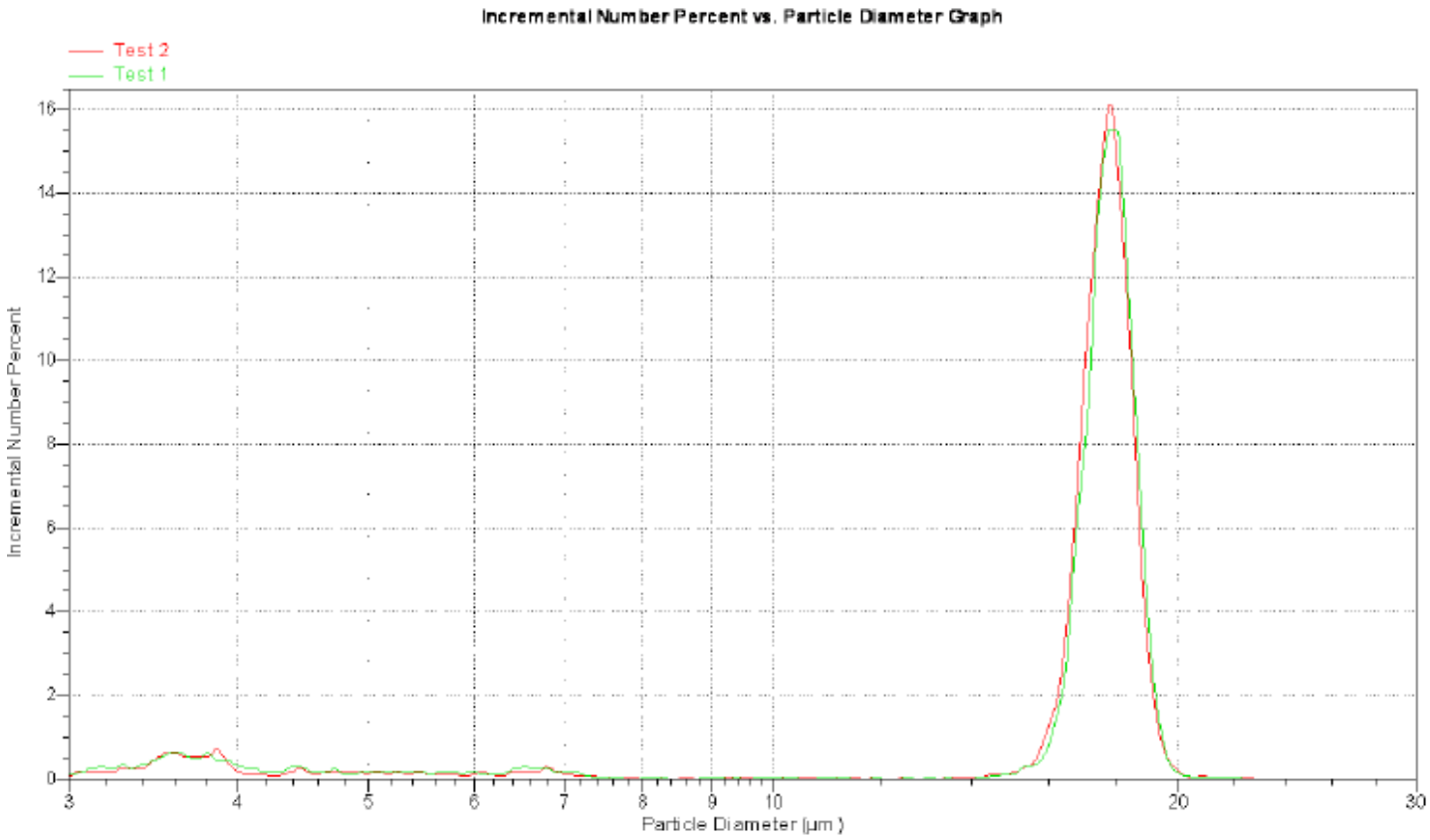
Applications

- Analysis of Grass Carp blood cells for additional chromosome, called a triploid, that indicates that the fish is sterile.
- Blood cells without the extra chromosome, called a diploid, indicate that the fish is not sterile and can reproduce in the wild.
- Grass Carp released for control of waterway weeds must be sterile to protect indigent species. Therefore all carp must be tested prior to release.

Ideal for:

- Rapid analyses mean that all fish can be tested in a short amount of time.
- Software optimized for 5 to 10 second analysis, with almost no time lost between samples.
- On-screen live display indicates whether "diploid" or "triploid" cells are present. Data can be captured in a report if needed.

Particle Size Distribution of Short Ragweed Pollen, 2 Analyses



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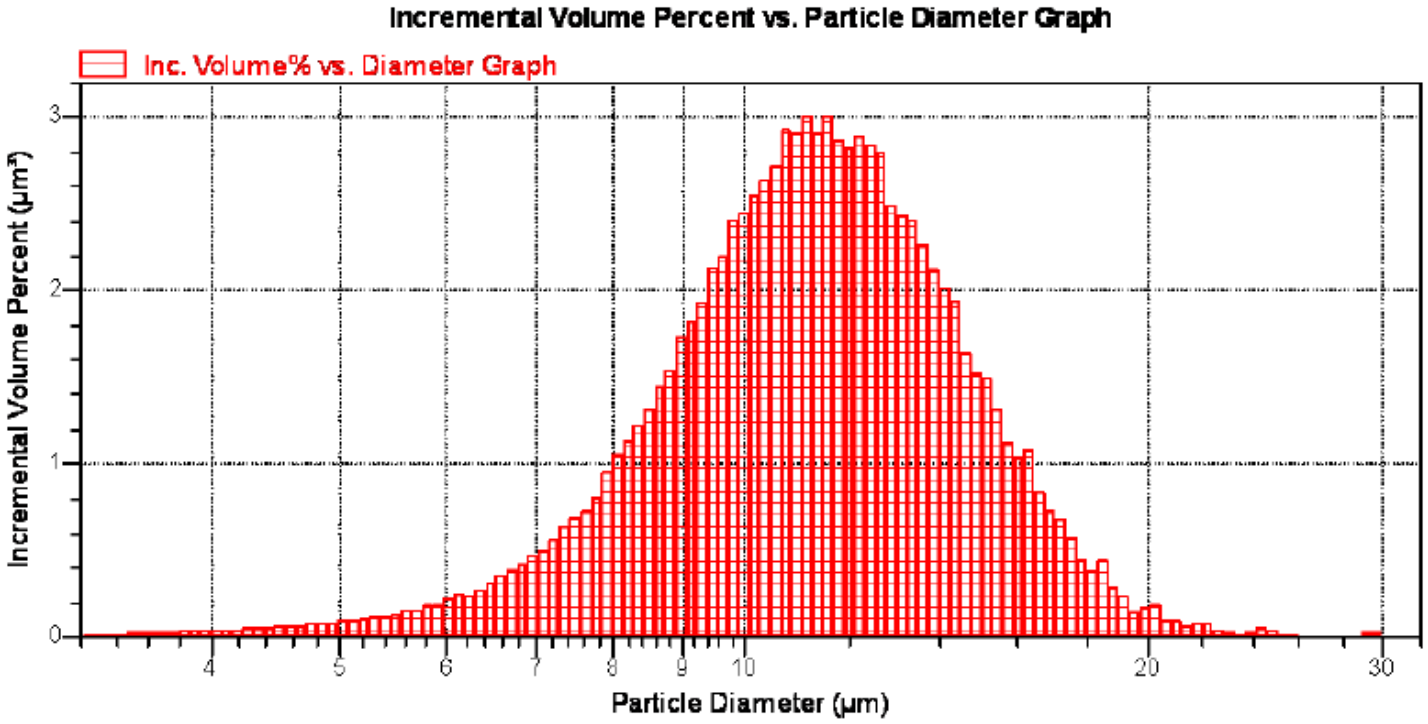
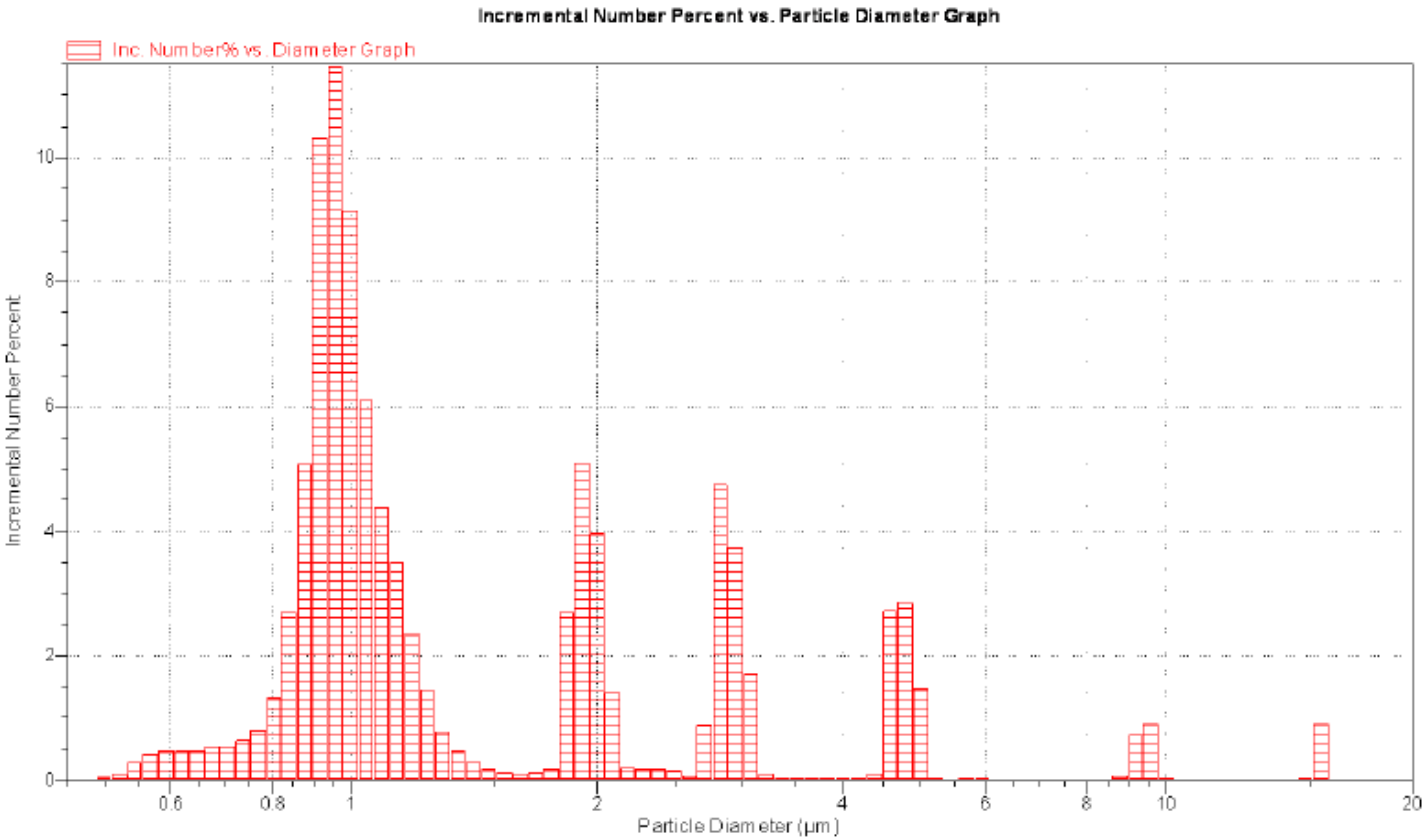
Particle Size Distribution of Coarse Garnet Sample

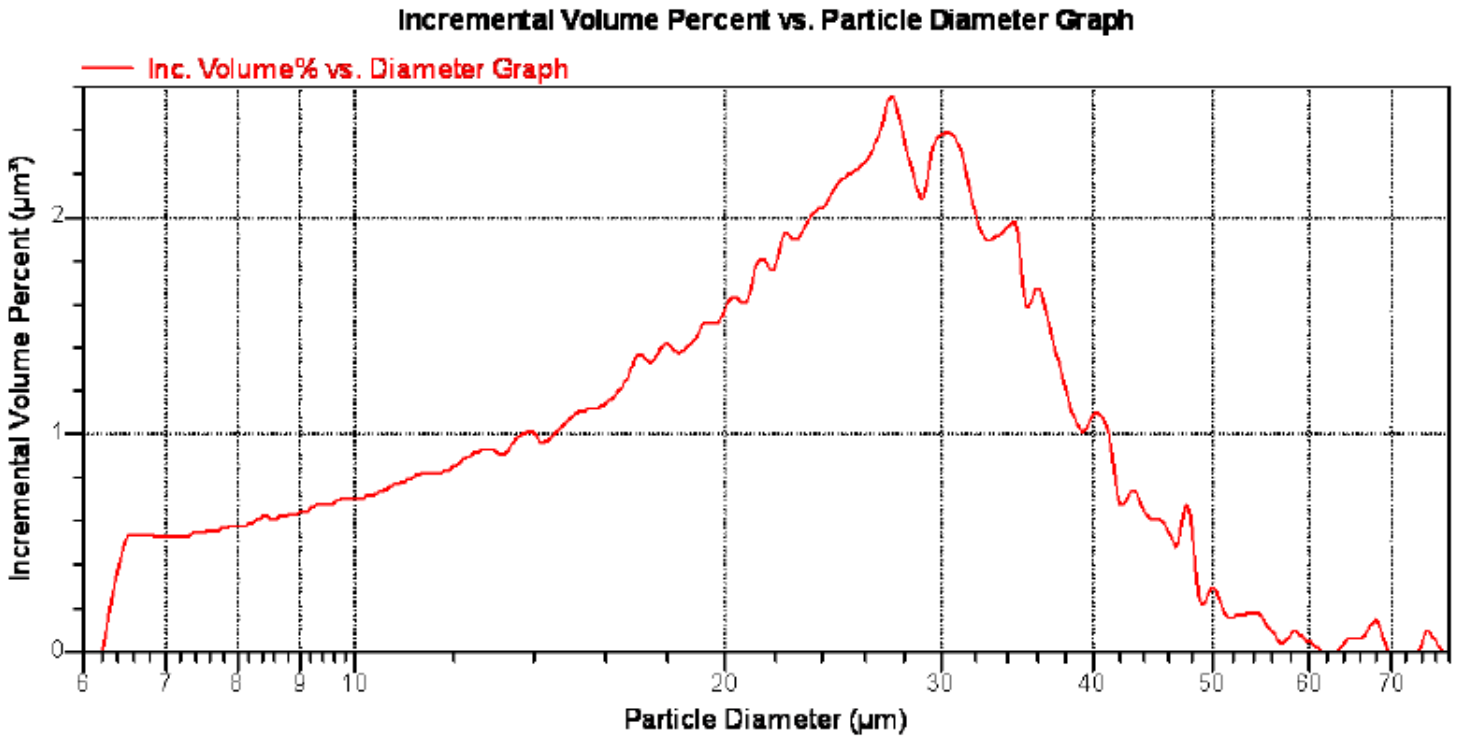


materials characterization WORKSHOP

Particle Size Distribution of Blend of Latex Spheres



Particle Size Distribution of Chocolate Powder – 190 µm orifice



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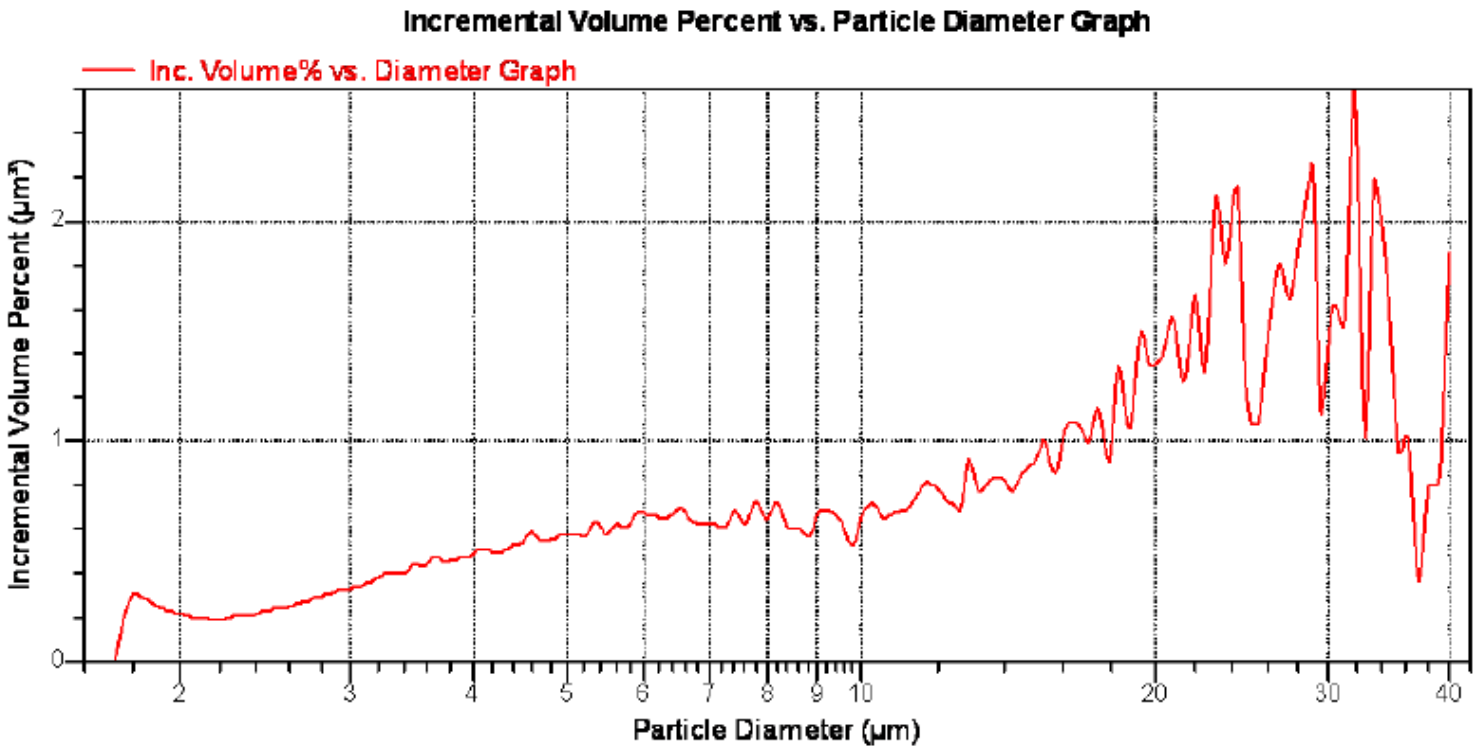
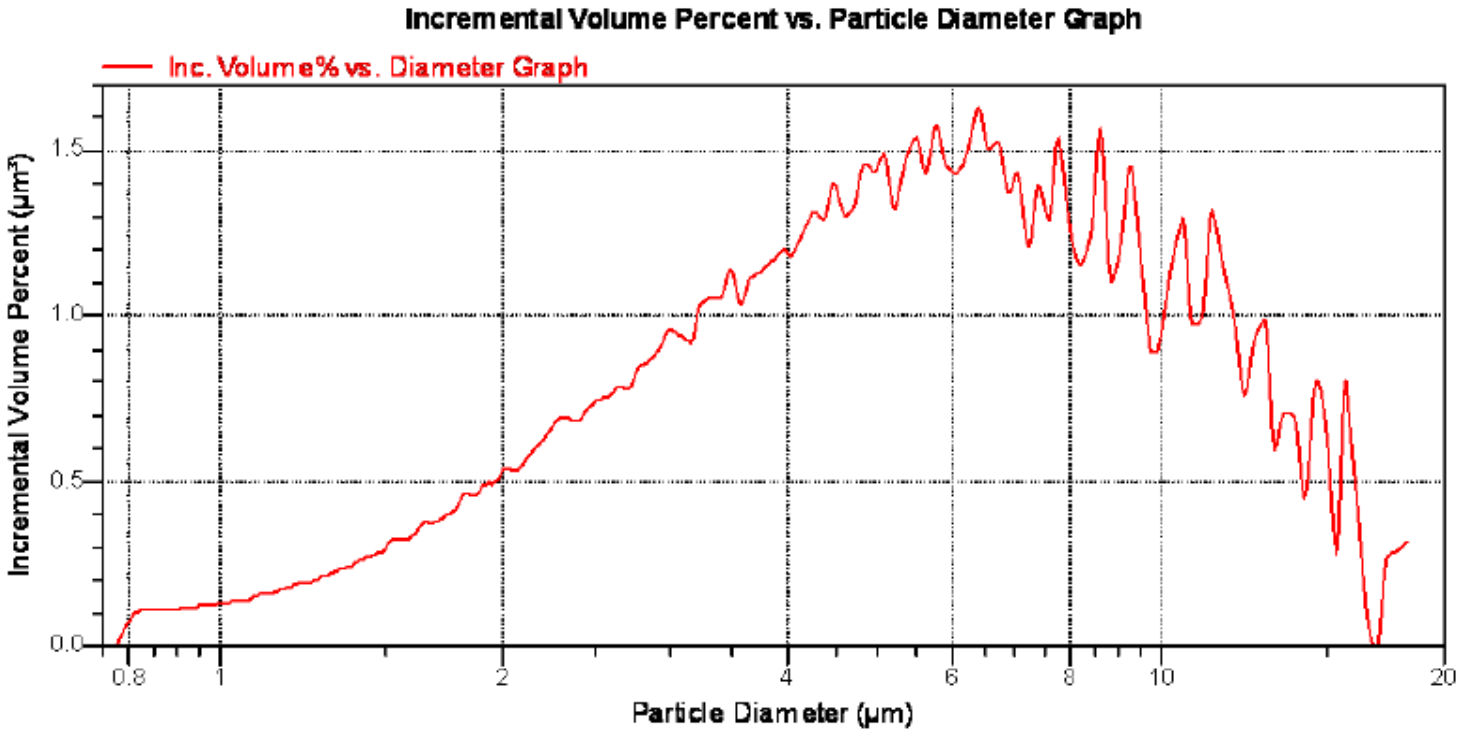
Particle Size Distribution of Chocolate Powder – 76 µm orifice



materials characterization WORKSHOP

Particle Size Distribution of Chocolate Powder – 30 µm orifice

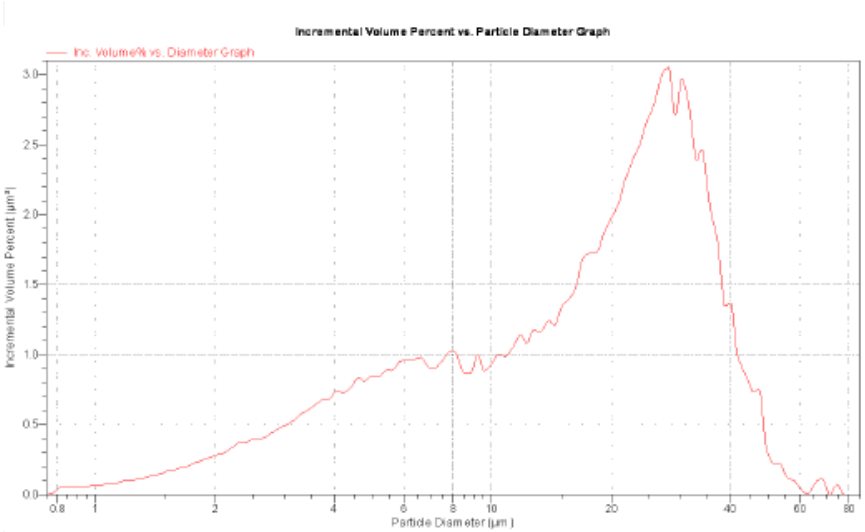


materials characterization WORKSHOP

Particle Size Distribution of Chocolate Powder – Blended Results

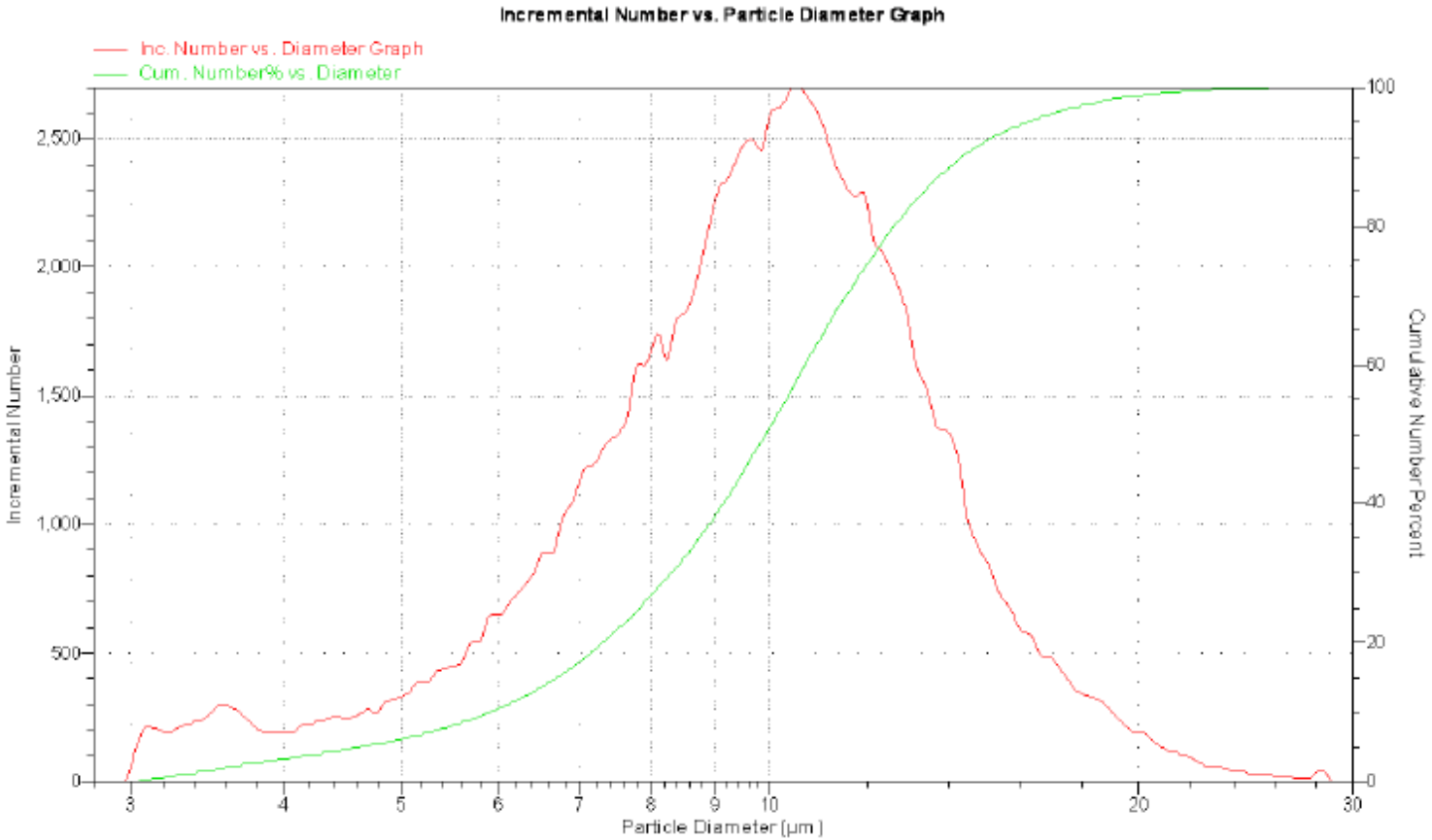
Blended Results

- 30:1 (diameter) dynamic range for each orifice tube.
- Full analysis using three orifice tubes – 190, 76, and 30 μm.
- Overlap of analysis range for adjacent tubes.
- Results of three analyses blended to single analysis.



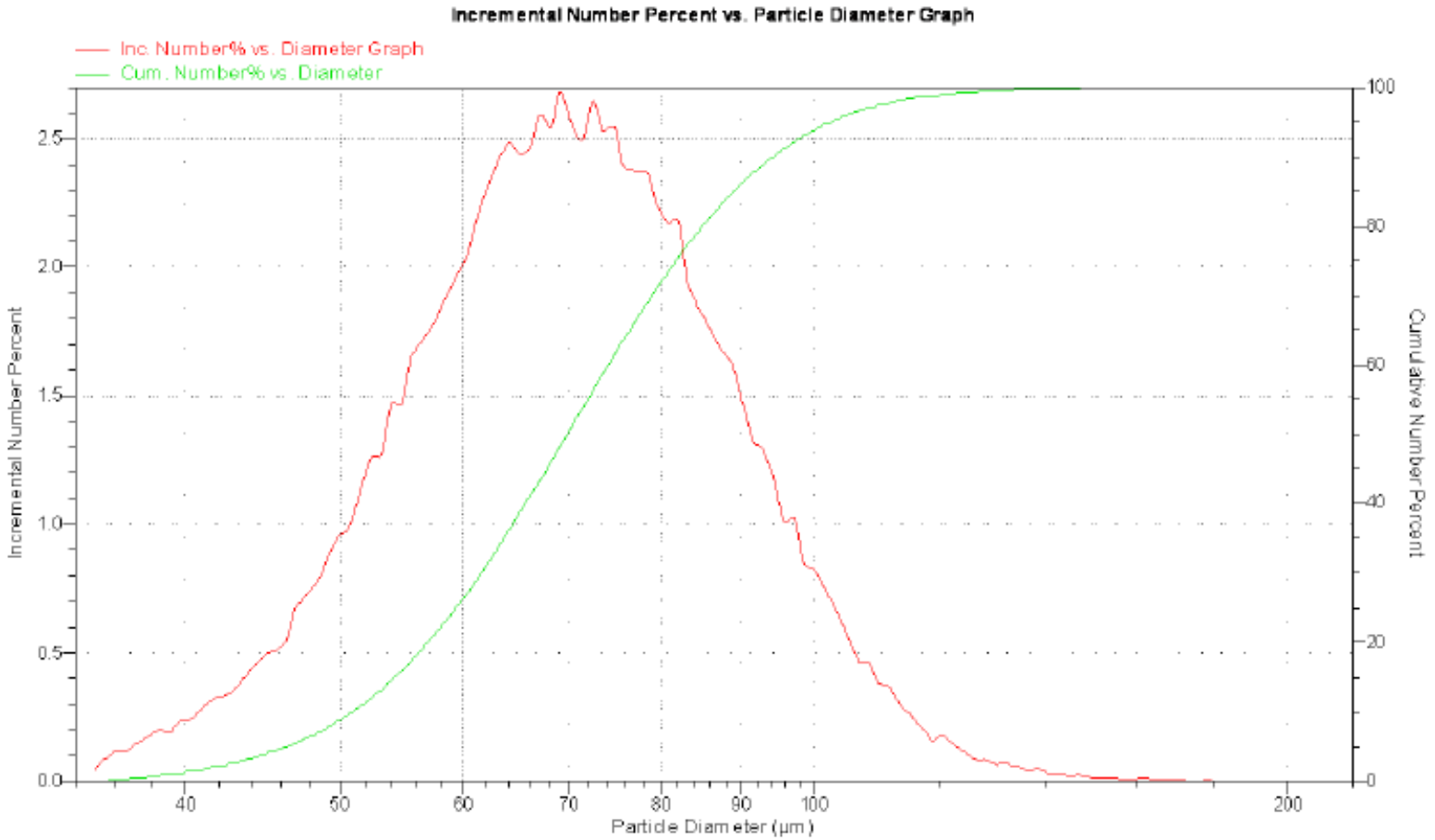
materials characterization WORKSHOP

Particle Size Distribution of Traceable BCR "Mirror" Standard

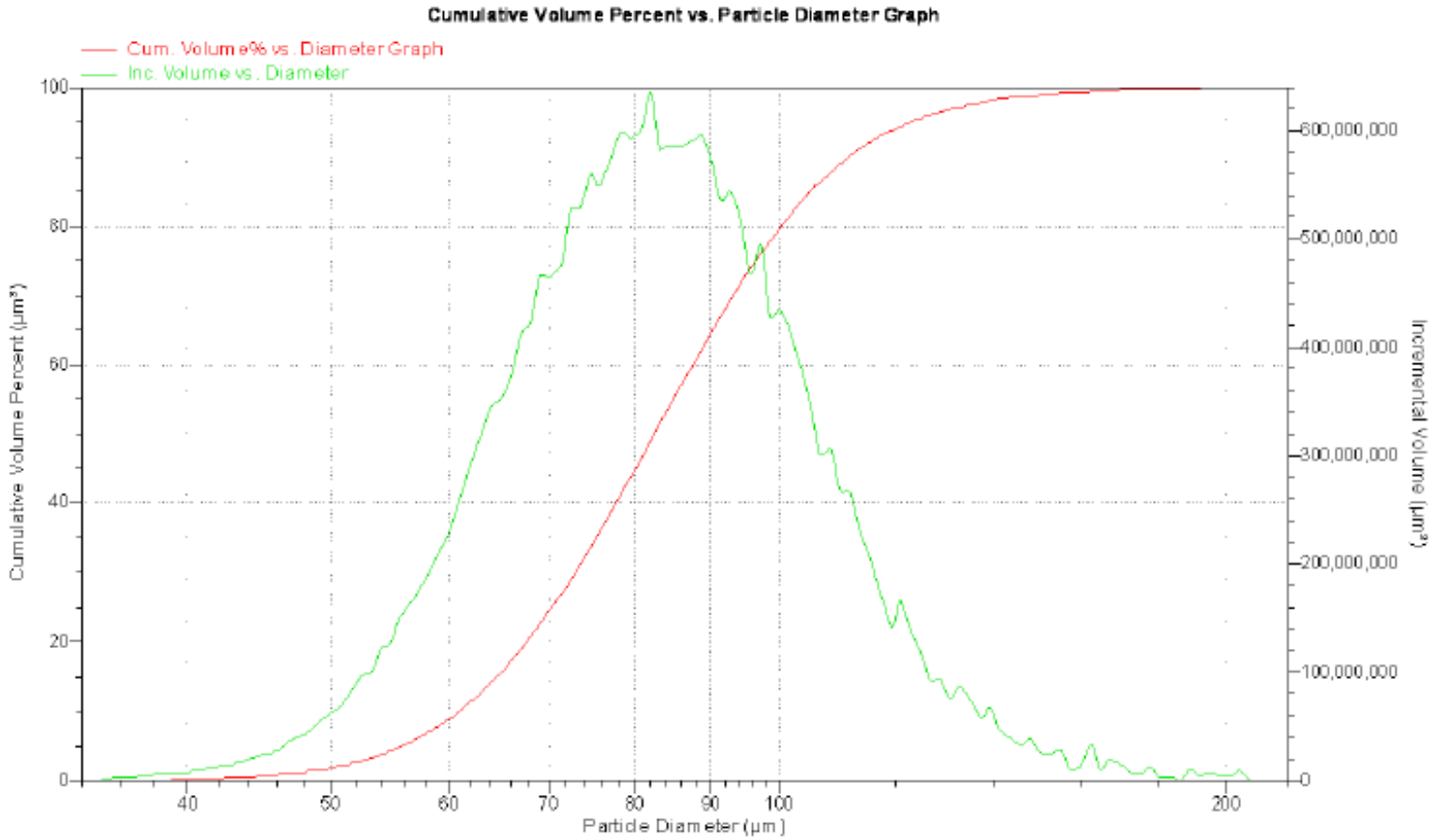


materials characterization WORKSHOP

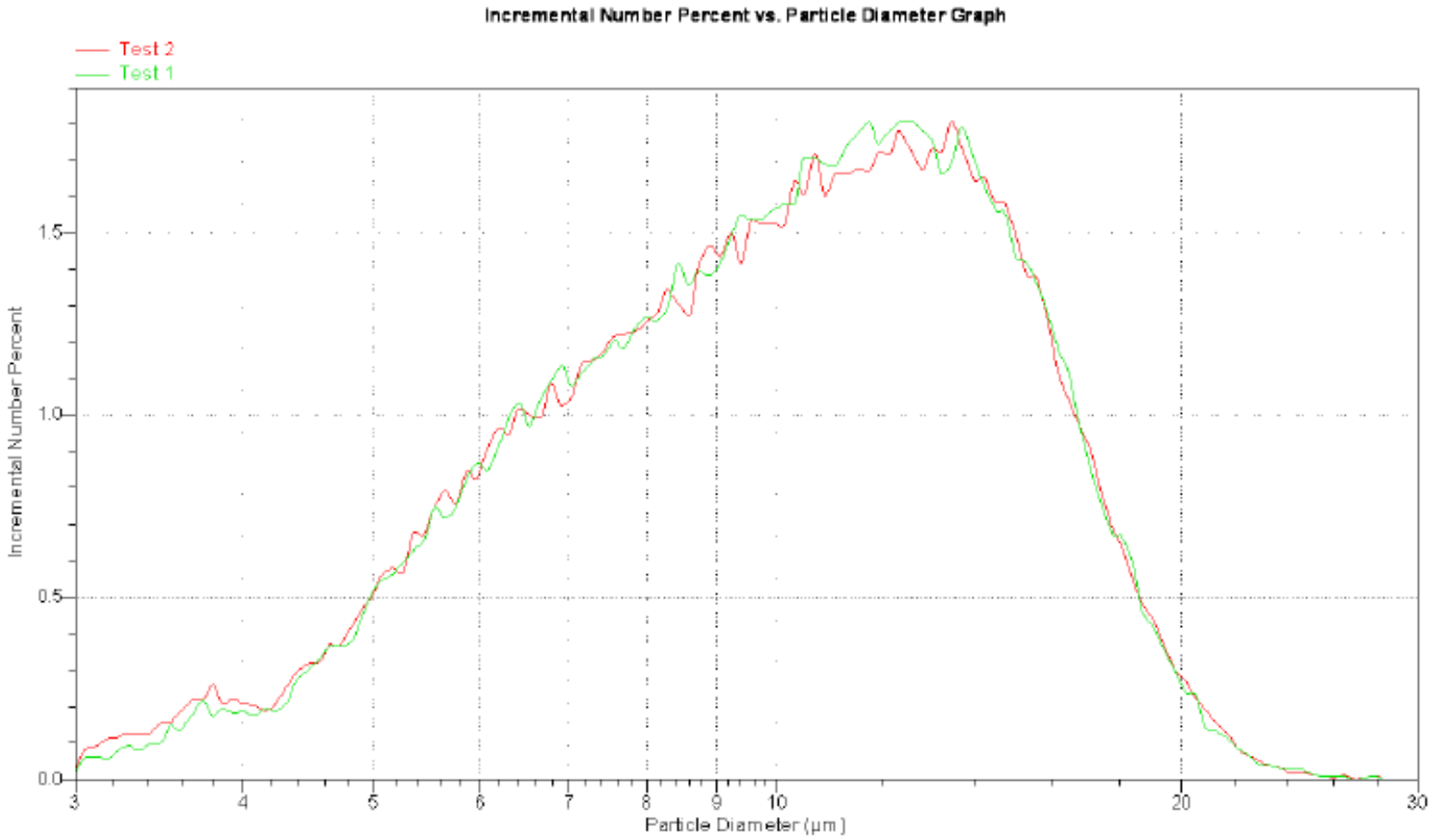
Particle Size Distribution of Alumina –Frequency and Cumulative Number Distribution



Particle Size Distribution of Alumina –Frequency and Cumulative Volume Distribution

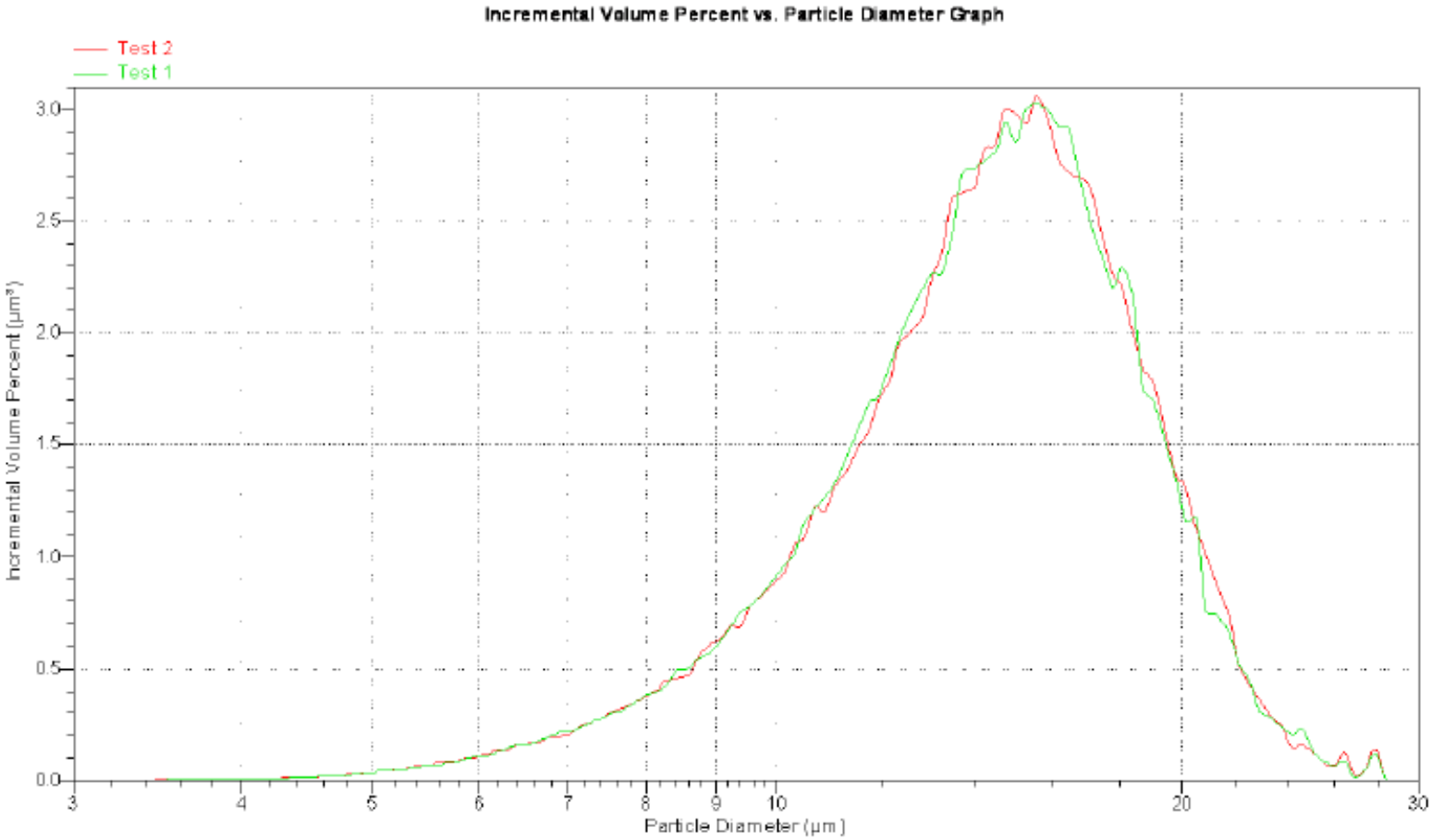


Particle Size Distribution of Corn Starch – Number Distribution, 2 Analyses



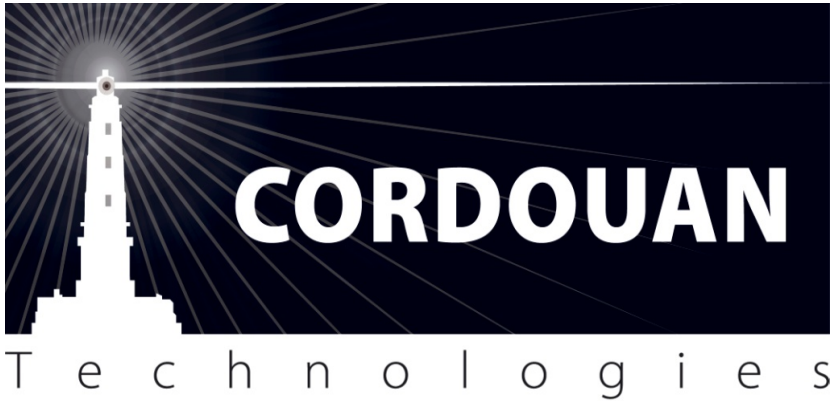
materials characterization WORKSHOP

Particle Size Distribution of Corn Starch – Number Volume, 2 Analyses



materials characterization WORKSHOP

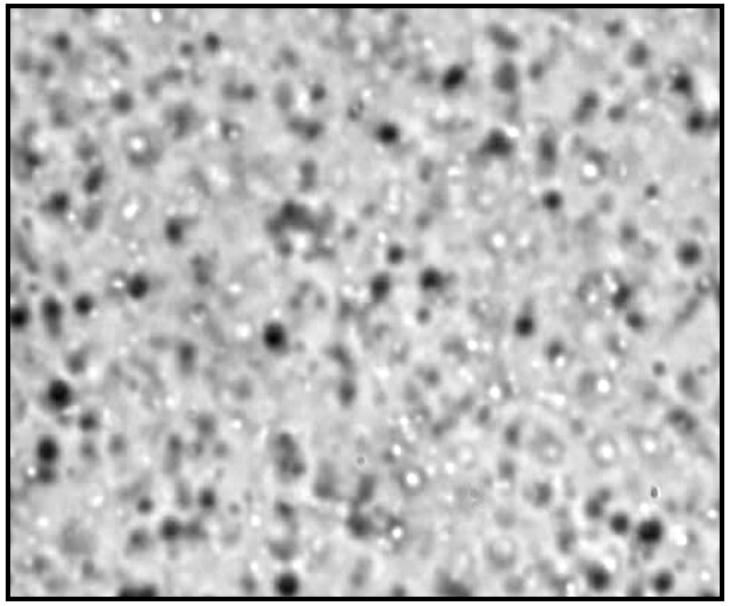
Particle size measurements of **dark** and **concentrated** dispersions by **dynamic light scattering**



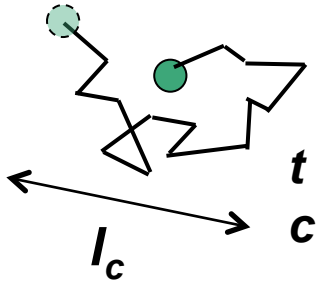
B. Maxit

How to measure the size of submicrometre particles.

Principle: The **Brownian motion** as a size signature



Observation of milk using optical microscopy



Diffusion coefficient:

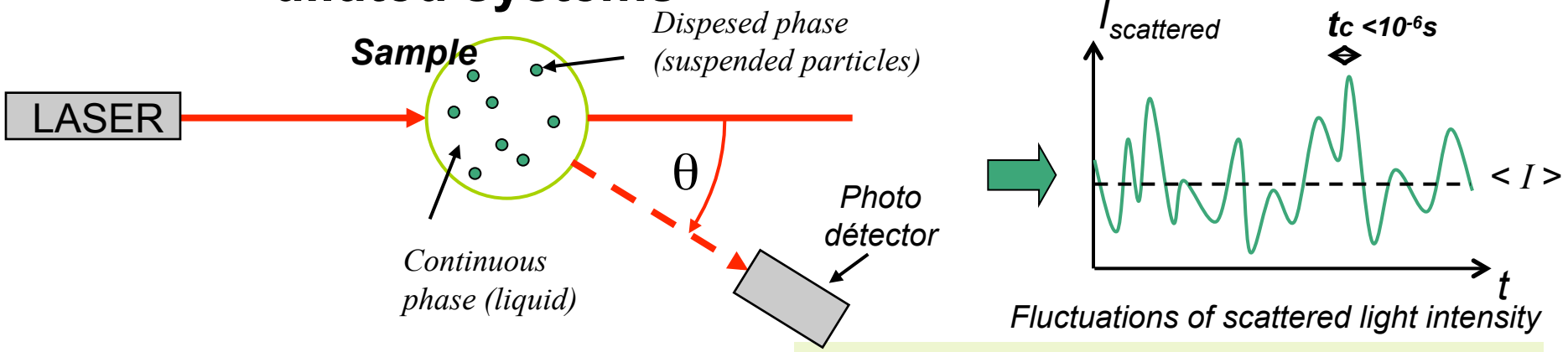
$D \sim l_c^2/t_c$

- Assumptions: spheres, without interactions, under brownian motion!

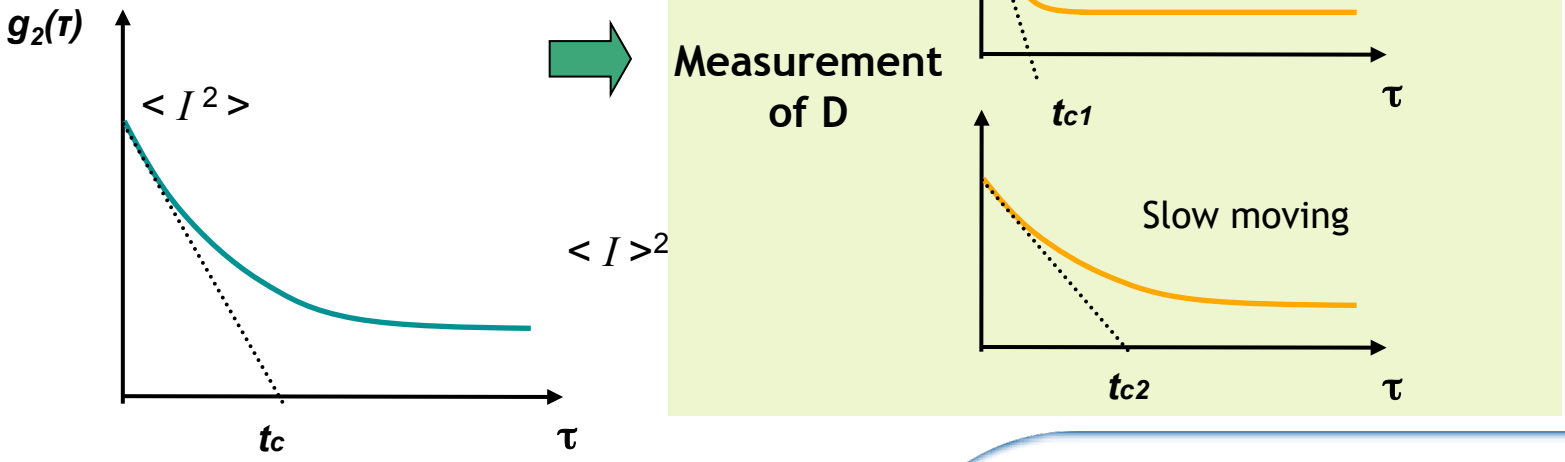
Stokes-Einstein $$D = \frac{K_b T}{6\pi\eta R_h}$$

- ***η Dynamic viscosity***
- ***R_h Hydrodynamic radius***

Principle of dynamic light scattering with diluted systems



- Self-correlation function



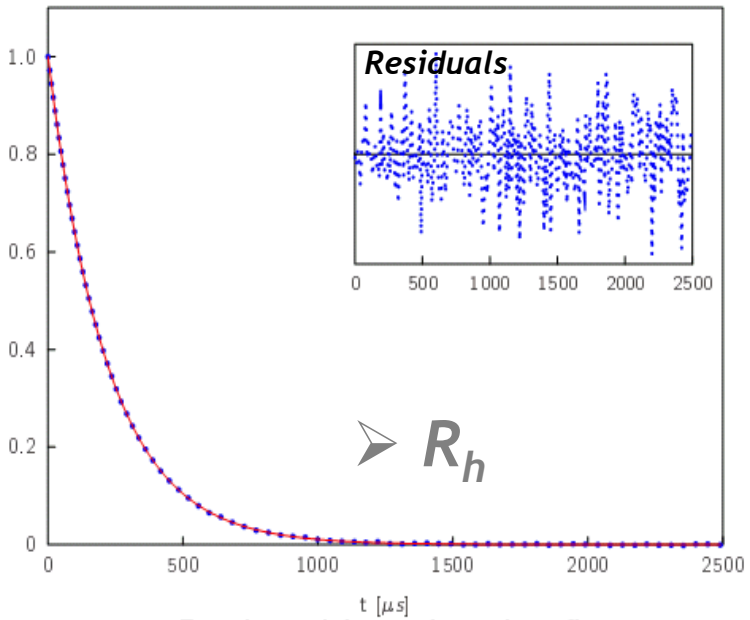
Autocorrelation function processing

Autocorrelation function

Monodisperse system

$g_2(\tau) \propto \exp(-2Dq^2\tau)$

➢ *Cumulants algorithm*



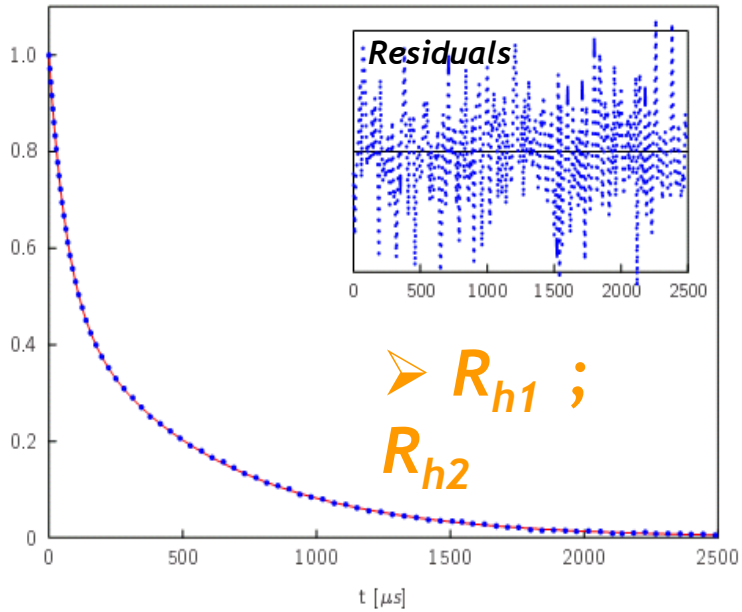
Experimental data and cumulants fit

➢ R_h

Polydisperse system

$g_2(\tau) \propto \sum A_i \exp(-2D_i q^2\tau)$

➢ *Padé-Laplace Algorithm*



Experimental data and Padé-Laplace fit

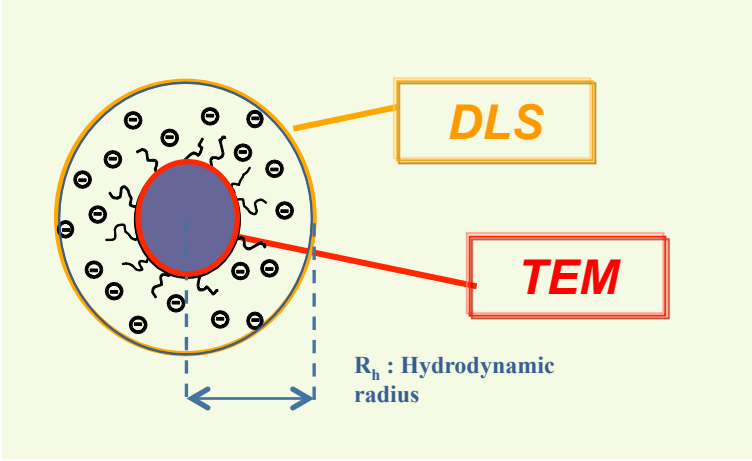
➢ R_{h1} ; R_{h2}

Dynamic light scattering with diluted systems

Main benefits of DLS:

- Measurement of the whole nanoparticle size range
- Often little or no sample preparation
- Statistical results in few minutes
- "Cheap" in comparison with other techniques

➢ **The hydrodynamic radius:**



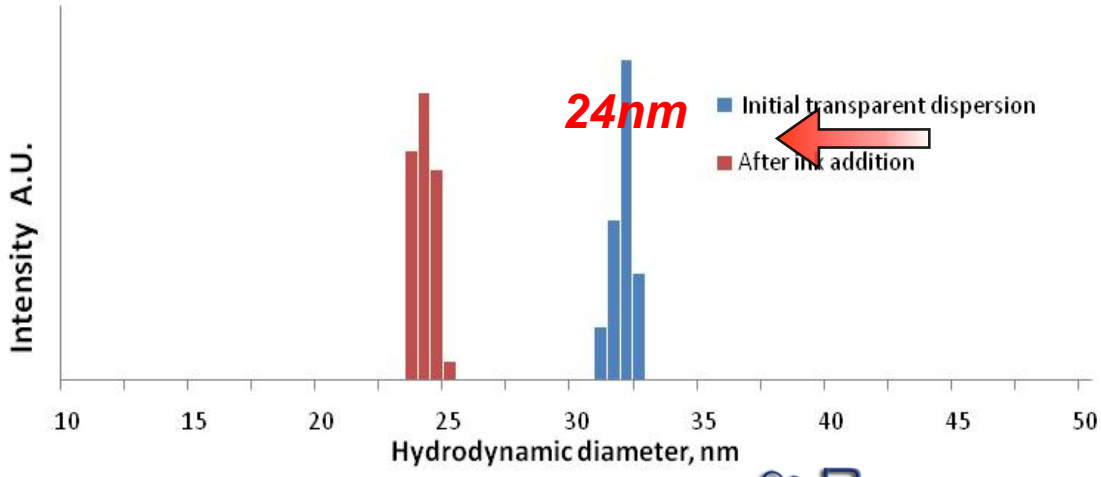
Traditional DLS limitations: dark/opaque and concentrated media

- Measurement of dark and opaque media

  - No signal

  - Local thermal effect induced by the laser beam (low heat capacity solvent/organic compounds)

*A standard polystyrene latex (Ø=**30nm** by TEM) is mixed with black soluble ink (10wt%).*



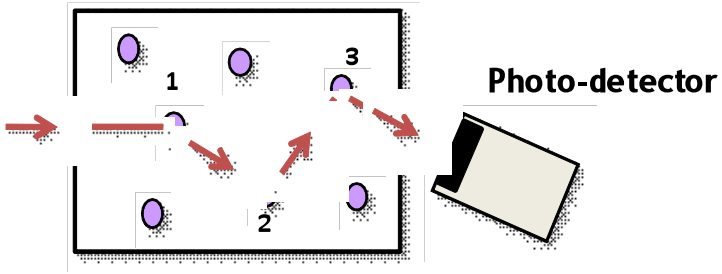
- ***25% error caused by the laser absorption!!***

Particulate Systems

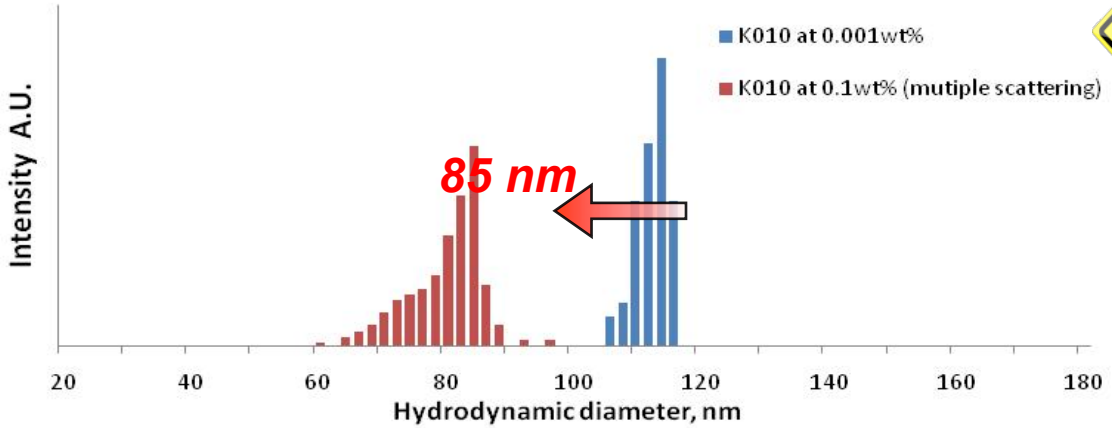
materials characterization WORKSHOP

Concentrated samples : multiple scattering effect

One detected photon correspond to N scattering events



*A standard polystyrene latex (Ø=**100nm** by TEM) measured at 0.1 wt %*

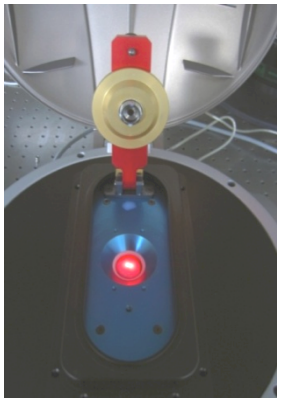


Artifact!!

- *Decrease of the measured hydrodynamic radius*

Solutions provided by the sample cell design

- An original design of the measuring cell
  IFP patent (EP 0654 661 A1)

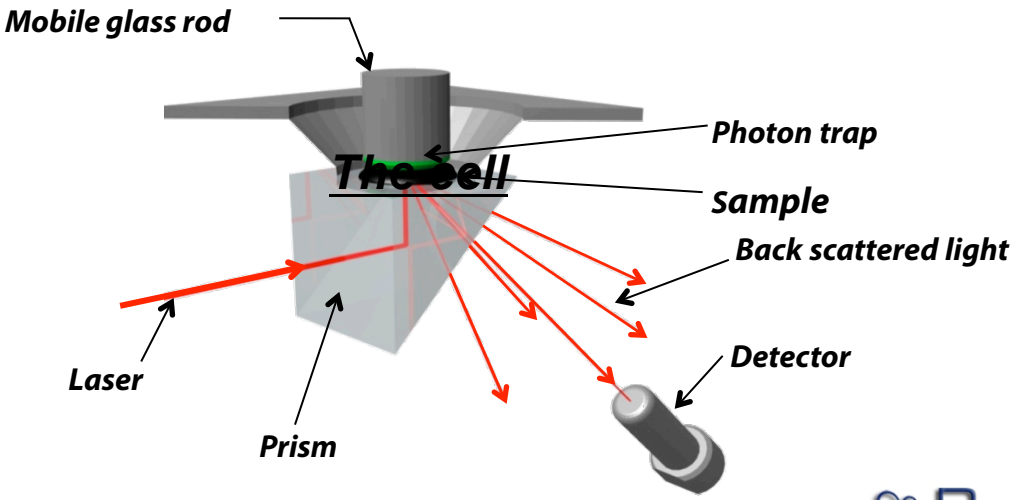


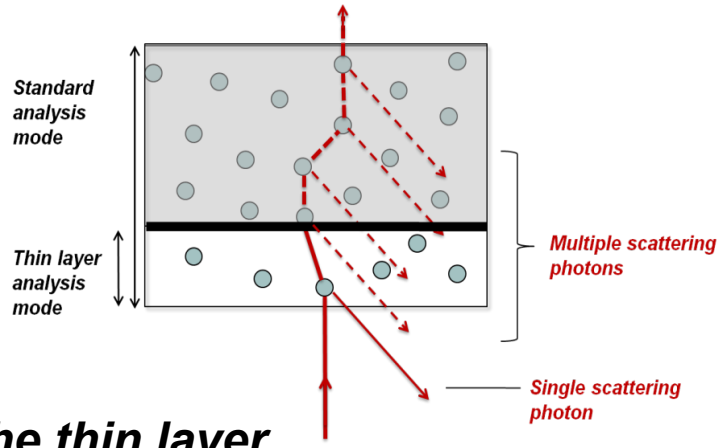
➢ ***Thin layer analysis:*** *prevents the sample from local heating and multiple-scattering*

➢ ***Photon trap:*** *absorption of the excess of transmitted light*

➢ ***Backscattered light detection*** *(at 135°): higher detection efficiency in opaque media.*

➢ *Solvant-proof cell without needing consumables*



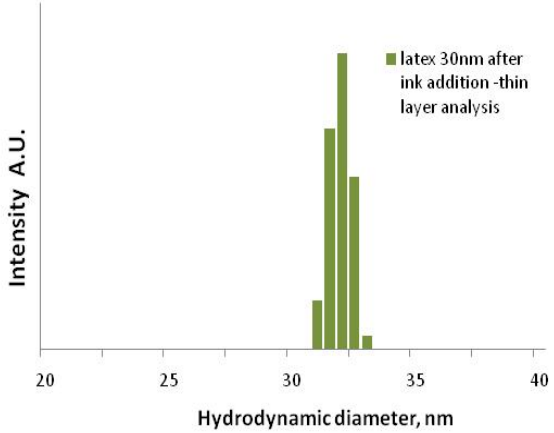


The thin layer analysis mode

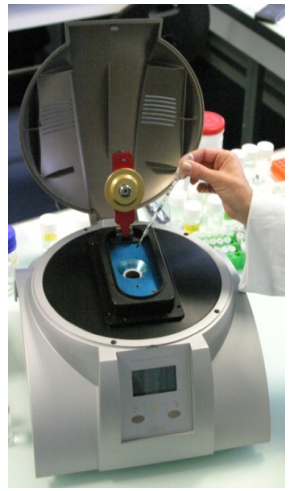
Solutions provided by the sample cell design

*Standard polystyrene latex (Ø=**30nm** by TEM) is mixed with black soluble ink (absorption).*

Thin layer measurement mode →

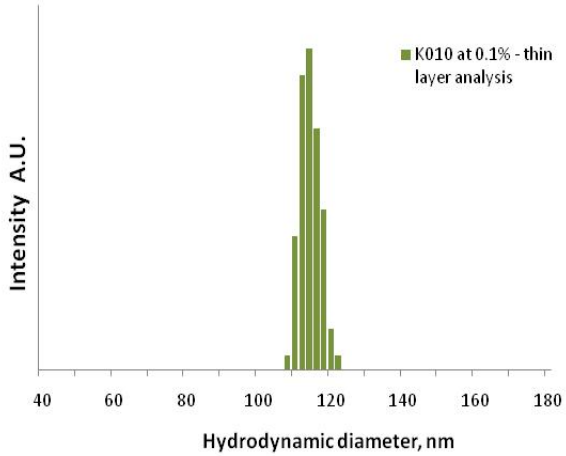


Hydrodynamic diameter = 32 nm



*Standard polystyrene latex (Ø=**100nm** by TEM) measured at 0.1 wt%*

Thin layer measurement mode→



Hydrodynamic diameter = 115 nm

Particulate Systems

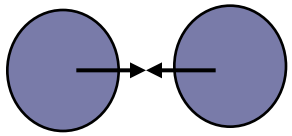
materials characterization WORKSHOP

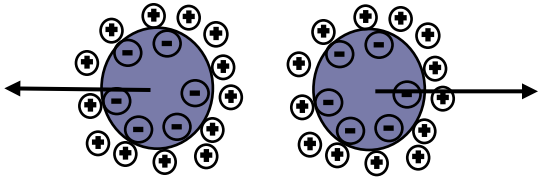
Concentrated samples : Interactions effects

- *A colloïdal system have to be generally stabilized against the attractive interactions caused by the Van der Waals forces*

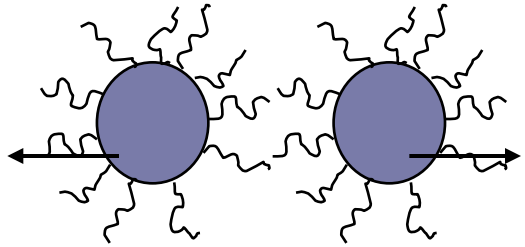
- *Stabilizing forces:*

Van der Waals



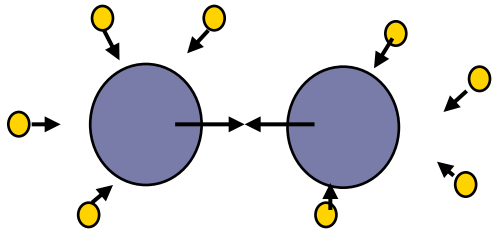


Electrostatic



Steric

- *Others destabilizing forces. Ex:*



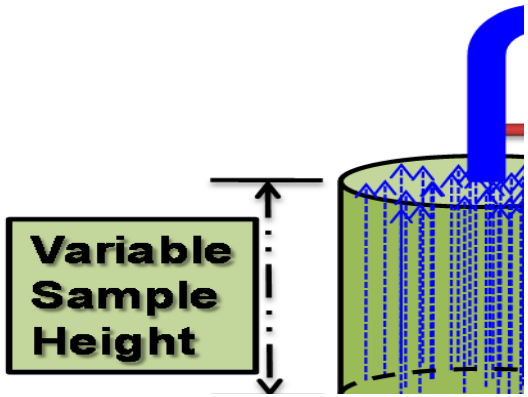
Depletion

➢ *These interactions will affect the motion of the dispersed phase !*

Air Permeability – Sub-sieve Auto-Sizer SAS from HEL

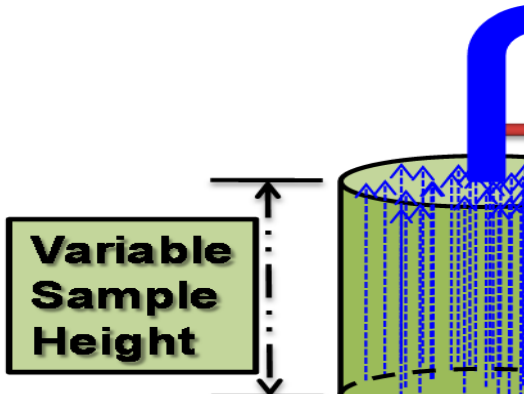
Air Permeability

- Developed as a replacement for the Fisher Model 95Sub-Sieve Sizer (FSSS), no longer available.
- Determines mean diameter for samples from 0.2 to 75 micrometers.
- Determines mean diameter based upon pressure drop across a bed of sample.
- Pressure drop is a function of external surface area of sample.
- Dry sample analysis only.



Air Permeability

- Applications include:
  - Pharmaceuticals
  - Paint
  - Toner
  - Geologic samples
  - Powdered Metals
- Significant work has been done with the pharmaceutical industry during development of this instrument.



Particle In-sight

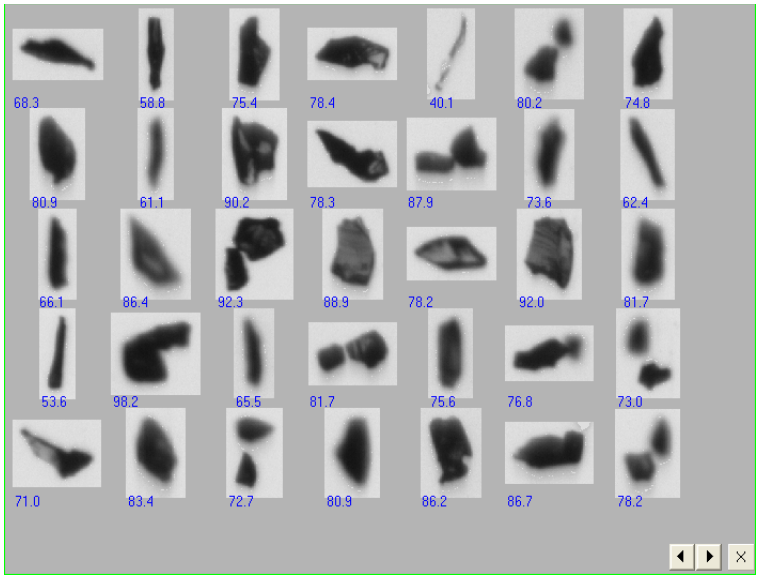
Particle Shape Analyzer

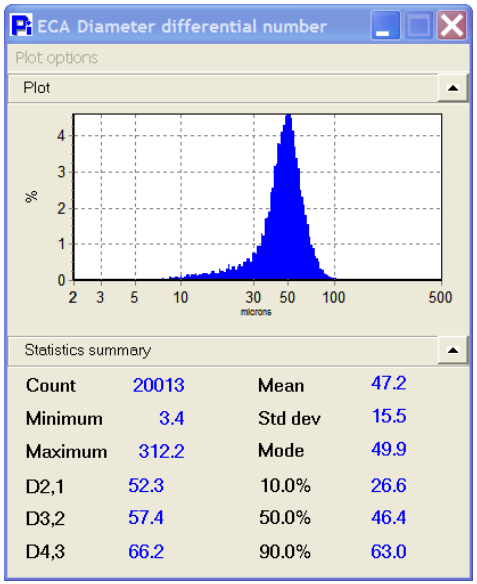
Particulate Systems

materials characterization WORKSHOP

Shape Matters

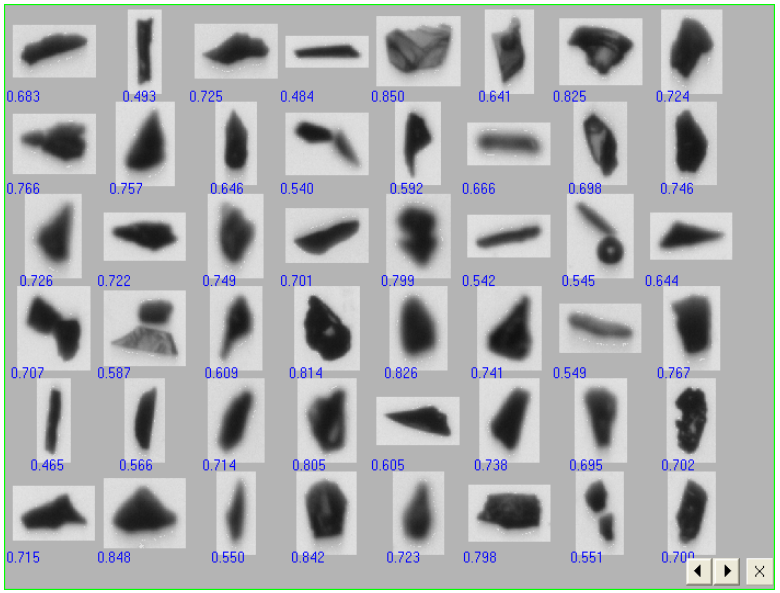
- In industry, characterization of particles plays a key role in product control.
  - Consistent size of pharmaceutical powders affects drug efficacy.
  - Size of cocoa affects the taste of popular chocolate products.
  - However, there is more to raw material quality control than just particle size.

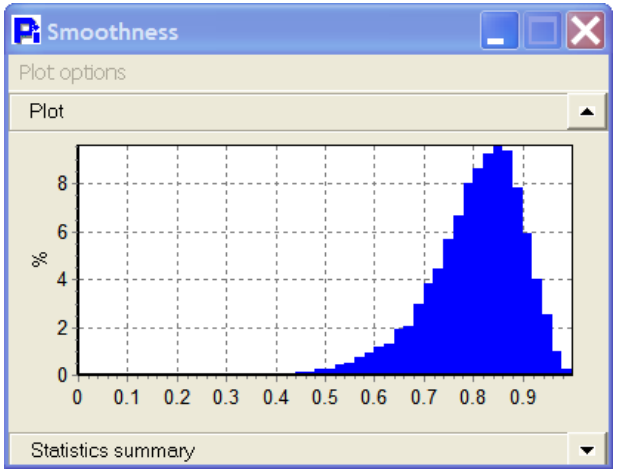




Shape Matters

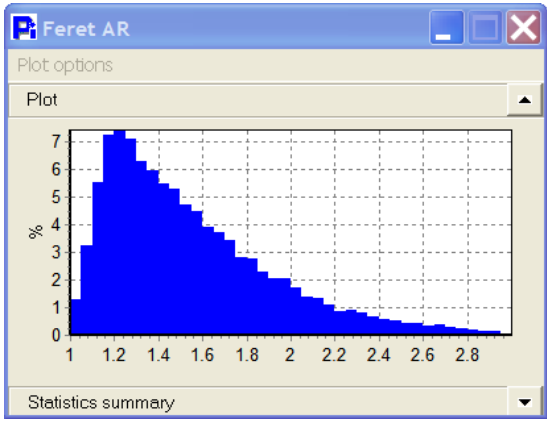
- Shape of particles has significant impact on quality of final many products.
  - Industries resorted to microscope-based analysis to control material shape.
  - These microscope-based methods are manual and non-quantitative in nature.
  - Basic microscopy is a very impractical method for quality control.

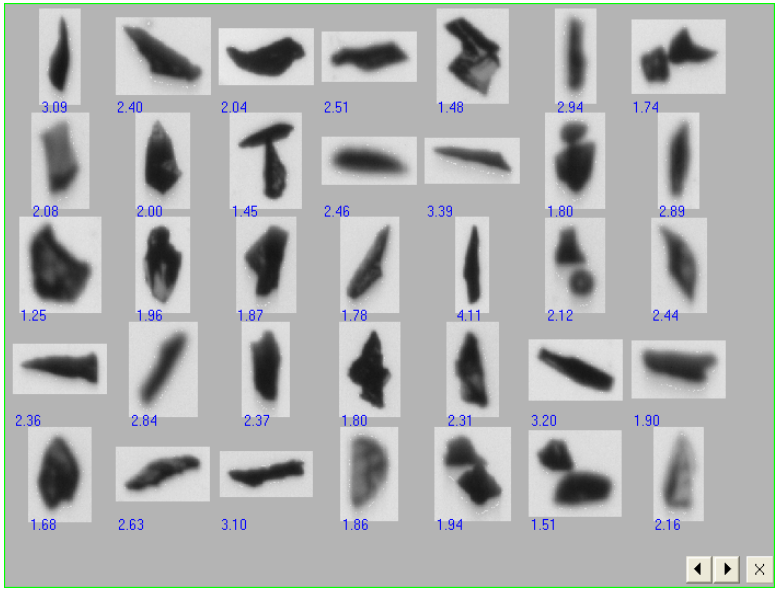




Shape Matters

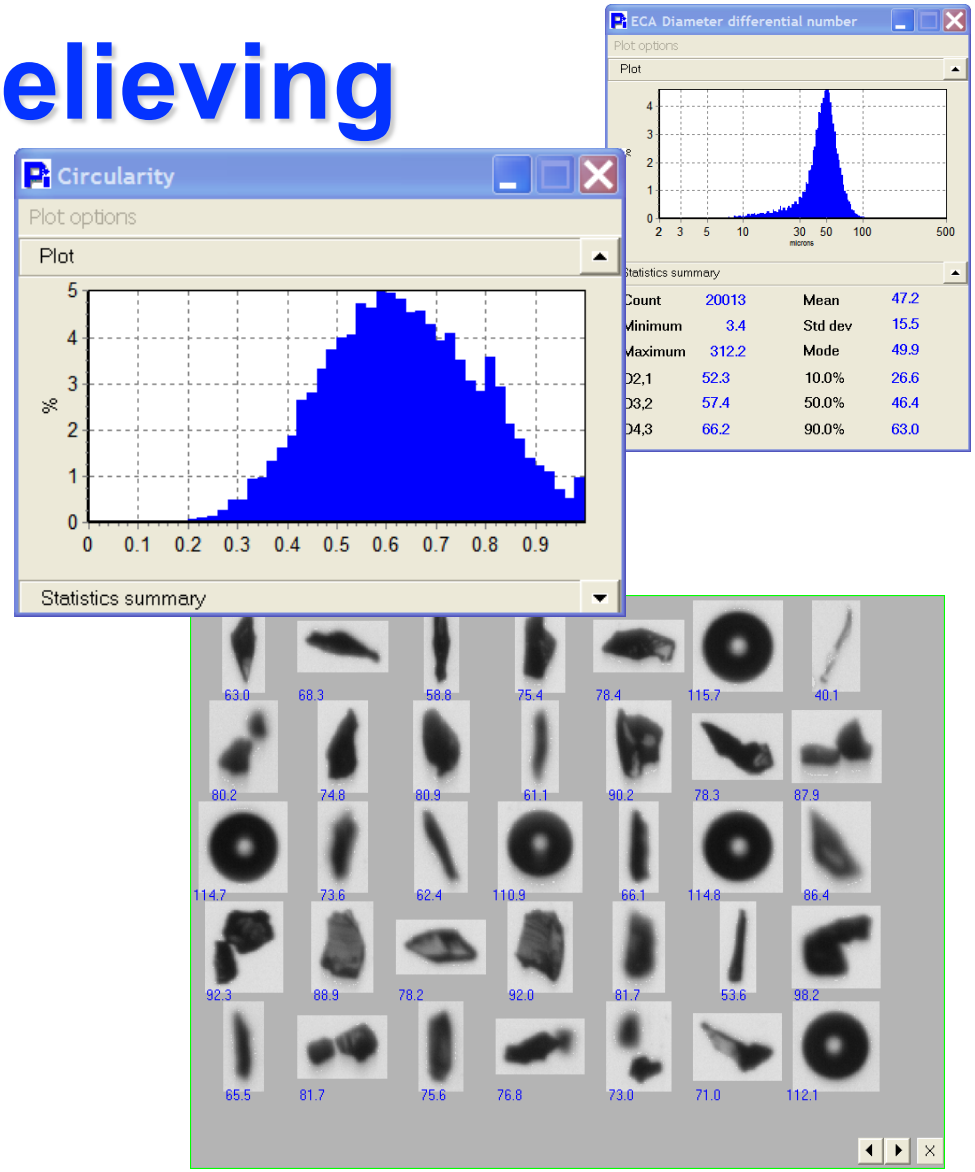
- Methods that only report a single size measure for a particle is inadequate for some applications where the nature of the raw material requires non-spherical (or exclusively spherical) geometry.
- In such applications a single diameter mean may not adequately characterize the particles.





Seeing is Believing

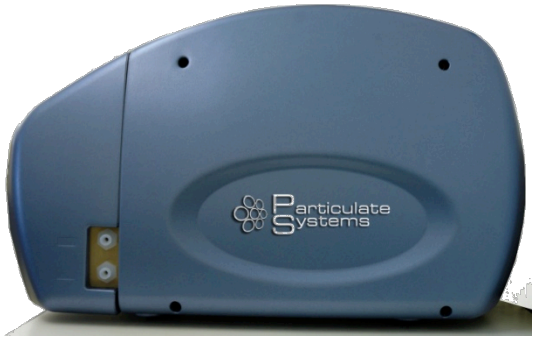
- Even when only the size of a specific material is needed, image analysis instruments offer additional confirmation of results.
- Typical image analysis systems can provide single-image size data of particles as well as captured images of the particles being analyzed.



Particle In-sight…What is it?

Dynamic Image Particle Shape Analyzer

- Key Features
- Speed of Analysis & High Resolution at High Magnification
- Comes standard with aqueous & organic capability.
- Post Run Particle Analysis.
- 3 to 300 micron measurement range
- 28 Shape Parameters
- ***Real time*** analysis and display
- 21CFR11 Compliant
- Recirculating system for statistical assurance. Single pass systems miss events.
- Save ALL particles analyzed.
- No limit to analysis.



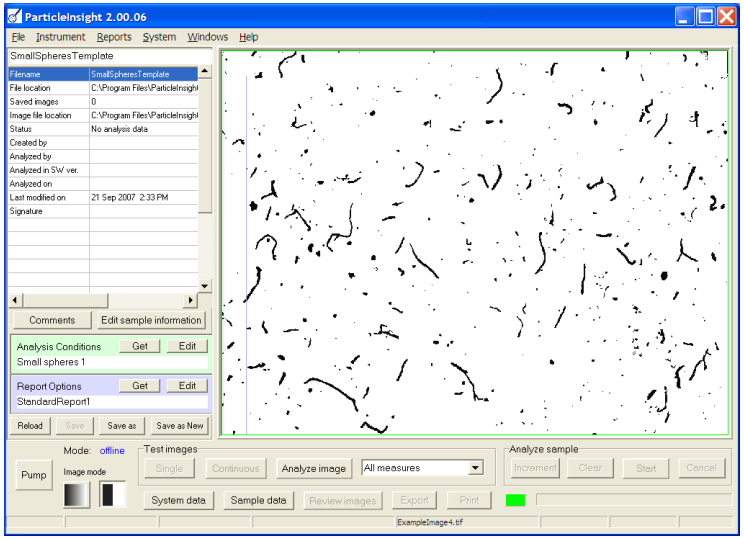


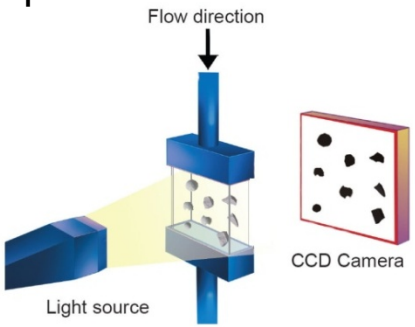
Image Analysis – derived from two perspectives

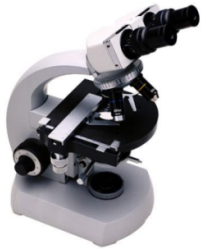
Dynamic Image Analysis

Evolved from a “Laser Diffraction” frame of mind.
Particles characterized while in motion.
Complies to ISO 13322-2.
Advantages:
Tends to ensure arbitrary orientation for better statistical representation.
Generally faster analysis.
Ease-of-use is the general focus.
Disadvantages:
Lower limit tends to be about 2µm due to optics.

Static Image Analysis

Evolved from a “Microscope” frame of mind.
Particles characterized while NOT in motion.
Complies to ISO 13322-1.
Advantages:
Due to limited depth of field (particles on a glass slide), the magnification can be greater and can analyze down to about 1µm.
Samples are analyzed in a dry form.
Disadvantages:
Usually smaller sample quantities measured.
Samples cannot be analyzed in a wet form.
Longer analysis time.
Care needs to be given to ensure adequate views of particles are analyzed.





Particulate Systems

materials characterization WORKSHOP

Particle In-sight – how it works

General diagram of the Re-circulating sample module:

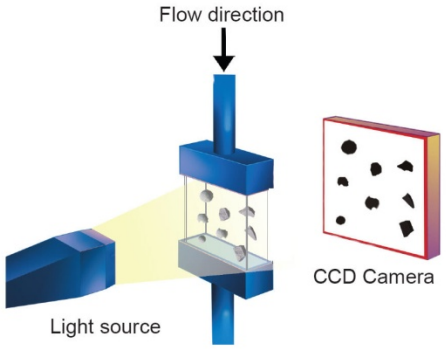
Built-in automated fluid transfer pump to fill system.

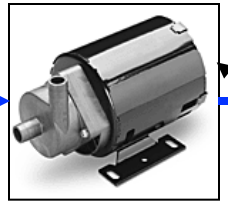
Sample Vessel with fill / drain and rinse capability and overflow control

Suspension Fluids. Aqueous or Solvents

Automated drain to waste

Analysis portion of system





Automated organic solvent compatible re-circulating pump

Superior Optics used in Particle In-sight

Telecentric Optics:

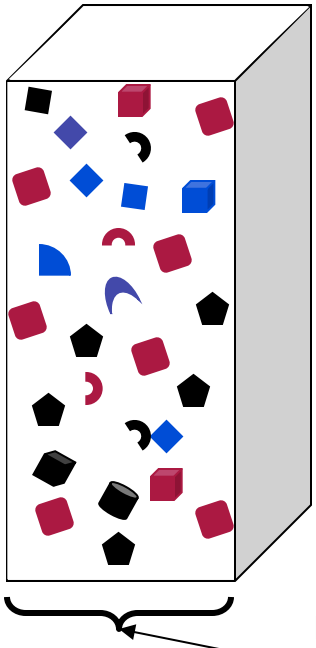
Depth of field in the analysis cell is greatly increased allowing for ALL the particles in the flow path to be in focus and analyzed (removes perspective errors).

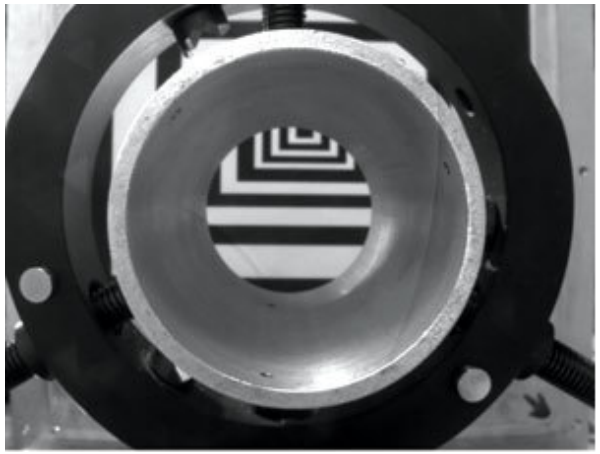
Benefits

•Better representative sampling and faster results due to a broad depth of field.

•Simple and robust design makes this less prone to failure.

•Added assurance of capturing rare events with Telecentric optics AND re-circulating system.





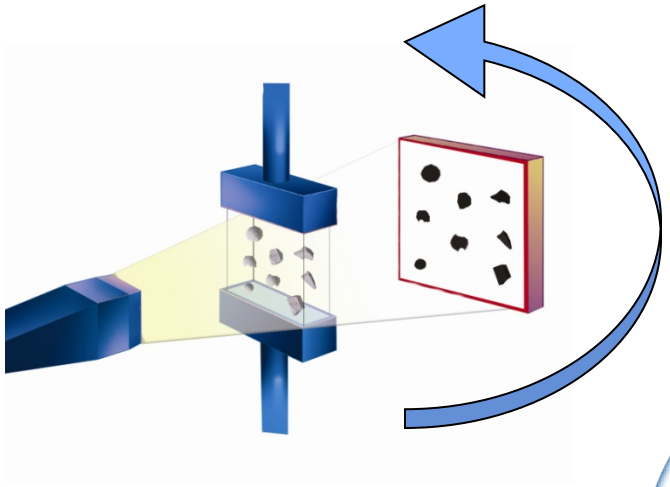
Opto Engineering

Particulate Systems

Particle Insight…..Depth of field is width of entire cell. A very broad depth of field means no lost events.

Recirculation versus Single-pass analysis

- Laser diffraction has employed sample recirculation for 40+ years to ensure a representative analysis of the population.
- Recirculating systems allow for re-analysis of the same aliquot. A must for process optimization.
- Given the dimension of the sensing zone and camera speed, it is highly unlikely to ensure each and every particle is captured in a single pass. Recirculation ensures full analysis of all particles.
- Single-pass systems generally use hydro-dynamic focusing that require expensive sheath fluids.



Random Orientation – a more representative view of particles

Random orientation ensures full analysis of particles. Controlled orientation will give just one view of your sample.

Laser diffraction has employed random orientation for 40+ years to ensure adequate characterization.

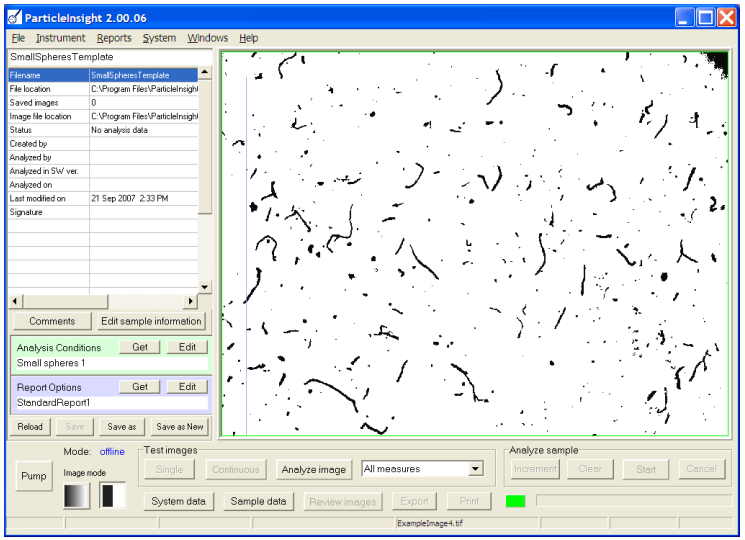
Static Image Analysis employs controlled orientation due to the Microscope-nature of the technique. Dynamic Image Analysis generally employs random orientation to ensure representative images and statistics are captured.

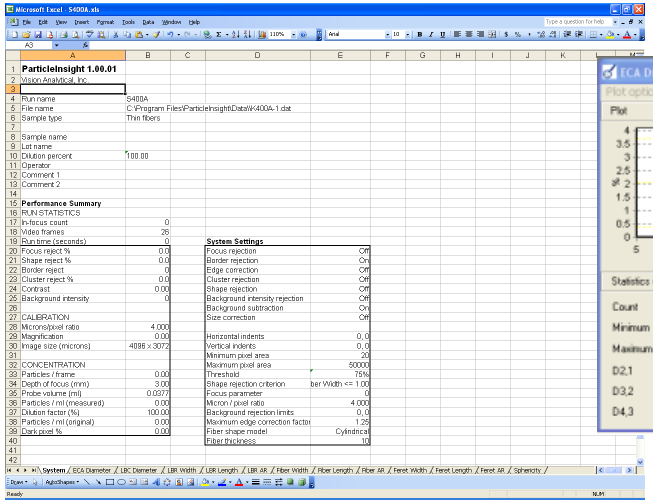
Random orientation of particles is cited Numerous times as a preferred (required) method

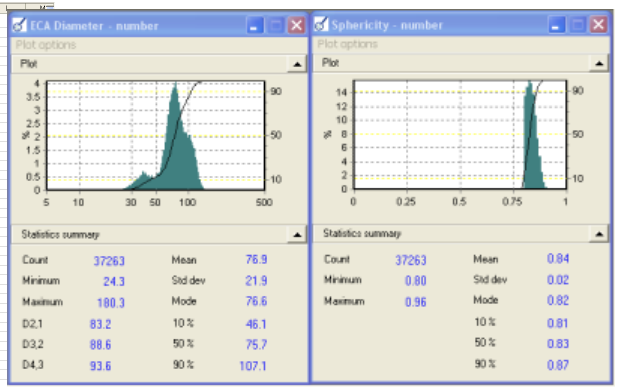
In this example, the exact same image (particle) is rotated to see random views of the particle. A controlled orientation device would only see ONE of these views rendering the unit useless to give the user critical information about his/her particle.

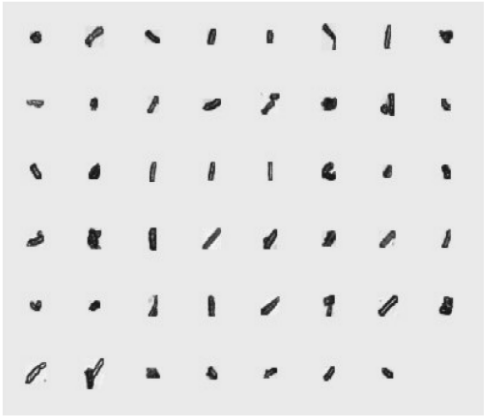
How it works (continued)

- Analysis extracts size and shape measurements for each particle using shape models.
- Statistical histogram counts updated as particles arrive.
- Particle statistics available for display, printing, or export to Microsoft XLS format. All Automatic done during analysis.



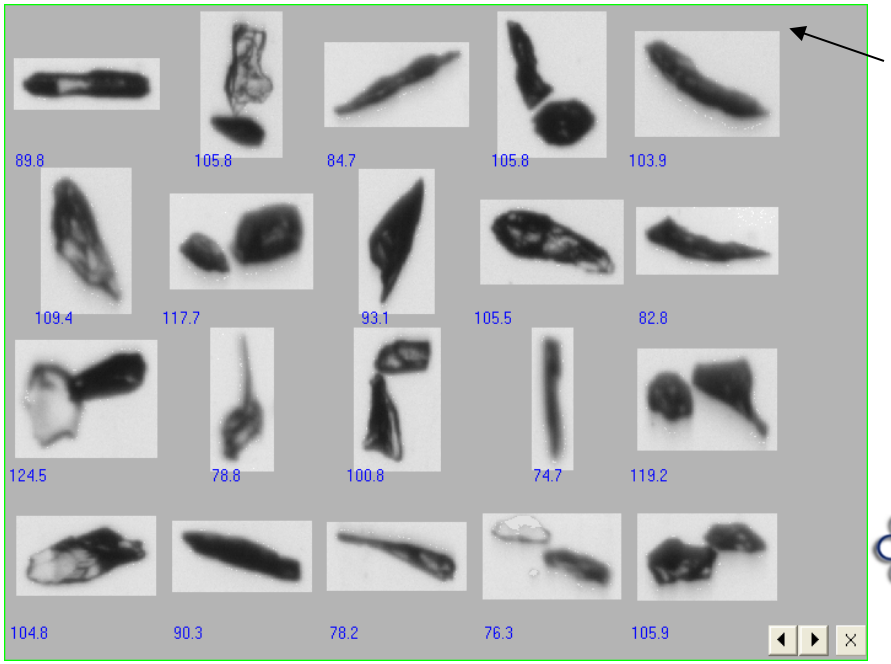






Capturing the “needle in the Haystack”

- Post Run Processing:
- All particle images are saved for review and re-analysis.
- Re-analysis can be done using any combination of the 28 available shape parameters.
- Particle Thumbnails also enables the statistical comparison between any two of the available 28 size/shape parameters.
- Accumulate simulated sieve data.



Particle Thumbnails

- Extracted particle from captured images that met user defined shape parameters.
- Each and every particle is captured during the analysis for post run processing

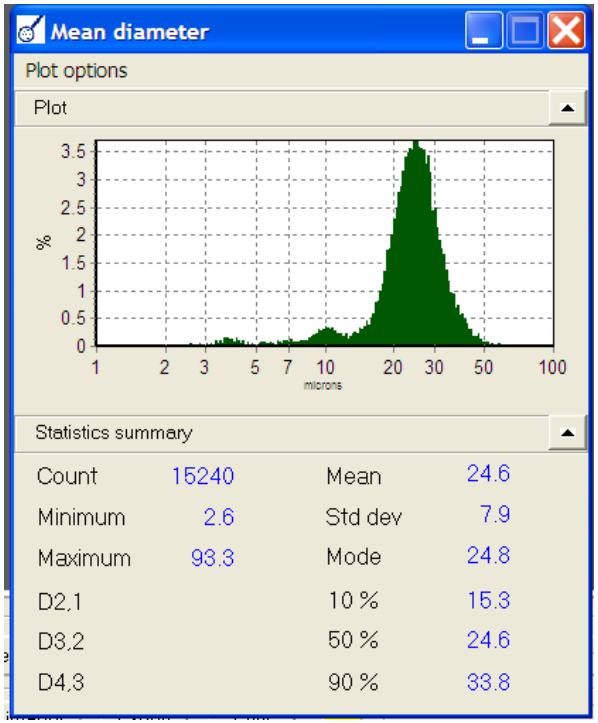
Screen Displays

Image display options:

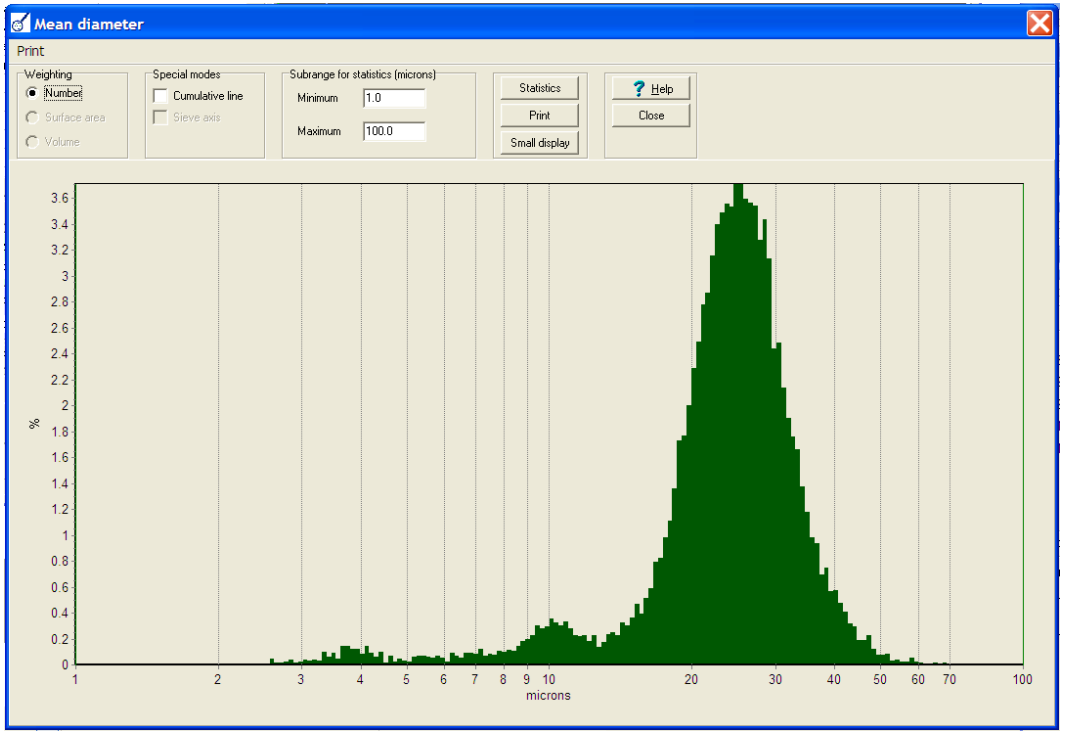
Full resolution window

Small data window with statistics

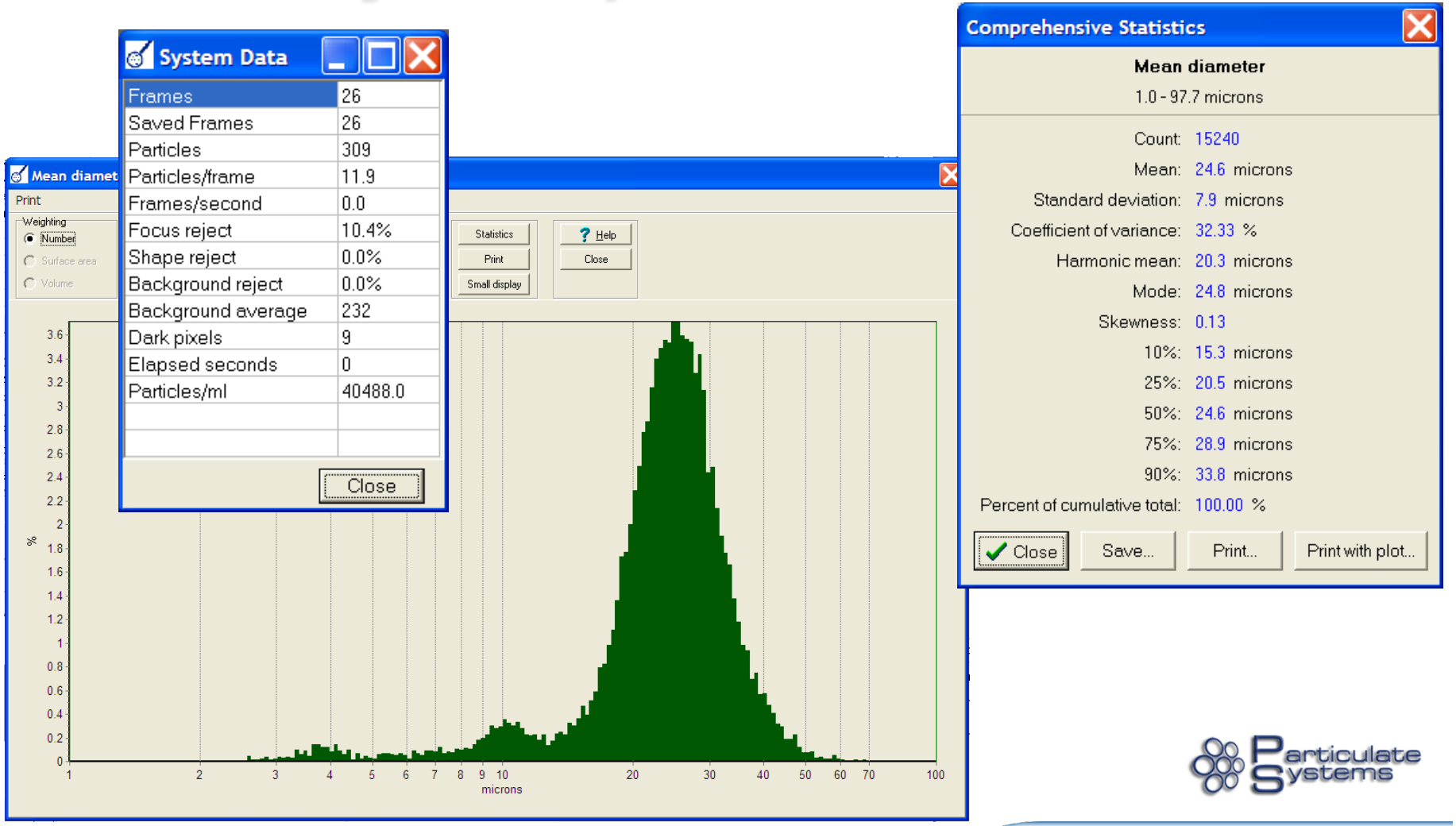
Small data window



Large data window

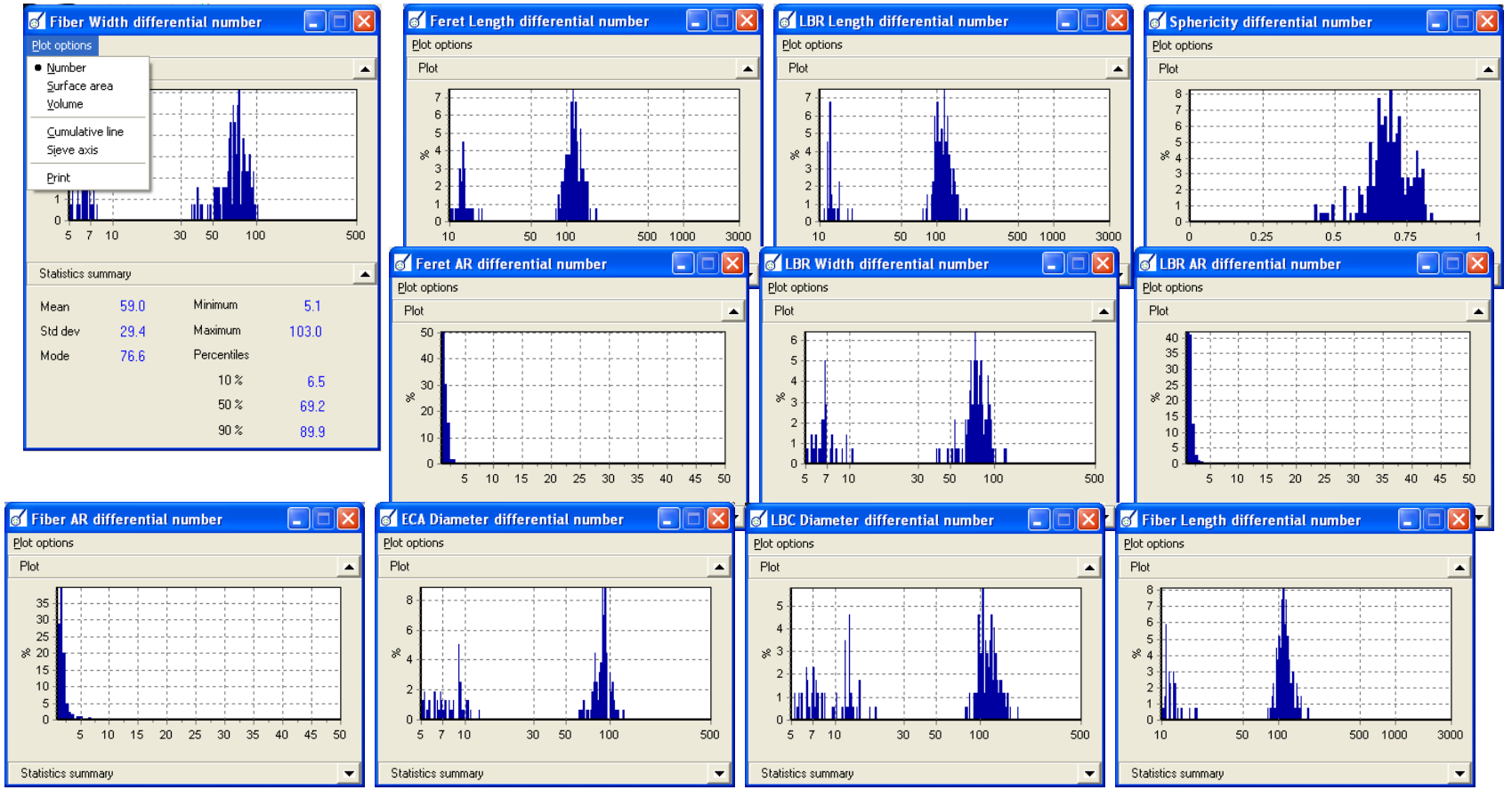


System performance results

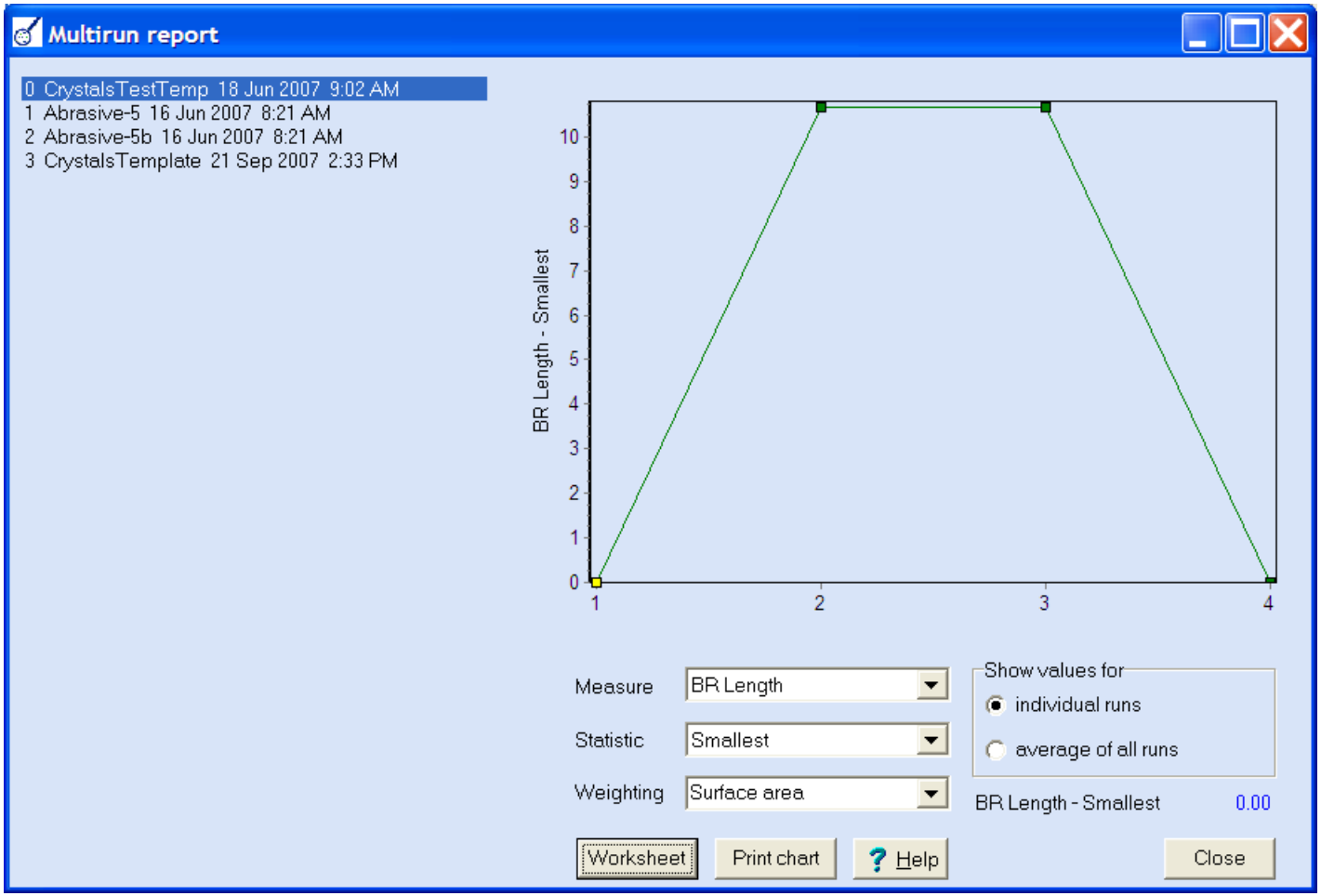


Flexible result presentations

- Data from an actual analysis of abrasive powders. More ways to report results via numerous measurement parameters enables better process quality control.



Keep track of you process with Sample Trending



- Multiple-analysis reporting feature allows averaging or trending of runs on any of the 28 available calculated measures.

Even more flexibility with Excel ...

- In the event that a user desires to make custom reports, other than those available with the Particle In-sight system, robust Microsoft® Excel® formats are available.

Microsoft Excel - S400A.xls

	A	B	C	D	E
1	**ParticleInsight 1.00.01**				
2	Vision Analytical, Inc.				
3					
4	Run name	S400A			
5	File name	C:\Program Files\ParticleInsight\Data\\K400A-1.dat			
6	Sample type	Thin fibers			
7					
8	Sample name				
9	Lot name				
10	Dilution percent	100.00			
11	Operator				
12	Comment 1				
13	Comment 2				
14					
15	**Performance Summary**				
16	RUN STATISTICS				
17	In-focus count	0			
18	Video frames	26			
19	Run time (seconds)	0		**System Settings**	
20	Focus reject %	0.0		Focus rejection	Off
21	Shape reject %	0.0		Border rejection	On
22	Border reject	0		Edge correction	Off
23	Cluster reject %	0.0		Cluster rejection	Off
24	Contrast	0.00		Shape rejection	Off
25	Background intensity	0		Background intensity rejection	Off
26				Background subtraction	On
27	CALIBRATION			Size correction	Off
28	Microns/pixel ratio	4.000			
29	Magnification	0.00		Horizontal indents	0, 0
30	Image size (microns)	4096 x 3072		Vertical indents	0, 0
31				Minimum pixel area	20
32	CONCENTRATION			Maximum pixel area	50000
33	Particles / frame	0.00		Threshold	75%
34	Depth of focus (mm)	3.00		Shape rejection criterion	ber Width <= 1.00
35	Probe volume (ml)	0.0377		Focus parameter	0
36	Particles / ml (measured)	0.00		Micron / pixel ratio	4.000
37	Dilution factor (%)	100.00		Background rejection limits	0, 0
38	Particles / ml (original)	0.00		Maximum edge correction factor	1.25
39	Dark pixel %	0.00		Fiber shape model	Cylindrical
40				Fiber thickness	10

System | ECA Diameter | LBC Diameter | LBR Width | LBR Length | LBR AR | Fiber Width | Fiber Length | Fiber AR | Feret Width | Feret Length | Feret AR | Sphericity

Ready NUM

Note one worksheet is automatically created for EVERY shape parameter the user selects for analysis. Each worksheet displays ALL the detailed results available for each parameter.

Efficient data reporting

Statistical data:

distribution histograms, weighted by number, surface area or volume
minimum value, maximum value, mean, standard deviation, mode
cumulative percentiles: 10%, 25%, 50%, 75%, 90%, by number, surface area or volume

Shape Models

Particle characterization shape models:

Circle: equivalent area (Heywood) diameter, equivalent perimeter diameter, bounding circle diameter, circularity, form factor, compactness

Ellipse: equivalent area diameter, bounding ellipse diameter, ellipsicity

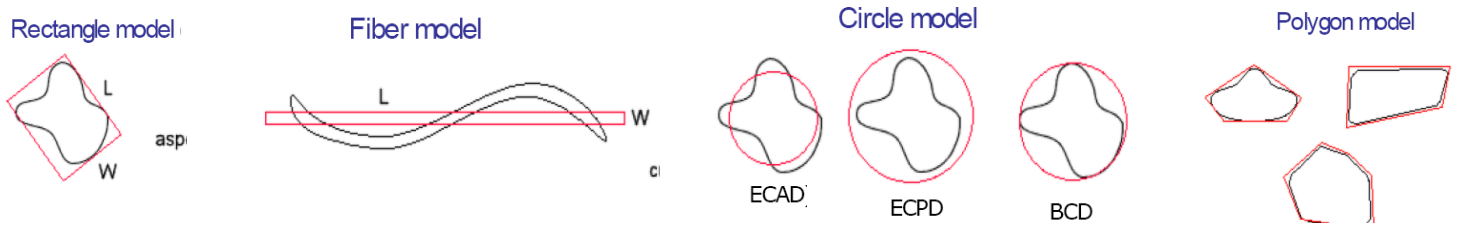
Rectangle: bounding rectangle length, width, aspect ratio; rectangularity

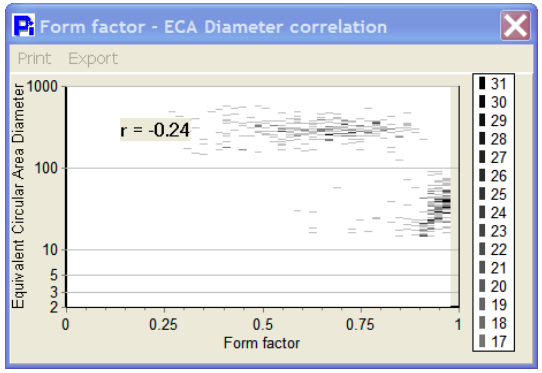
Polygon: polygon order, convexity

Fiber: length, width, aspect ratio, curl

Irregular: Feret length, width, aspect ratio, mean radius, smoothness

PLUS, ability to correlate any two using a Pearson coefficient.





Total of 28 Shape Parameters offered.

Particle In-sight™

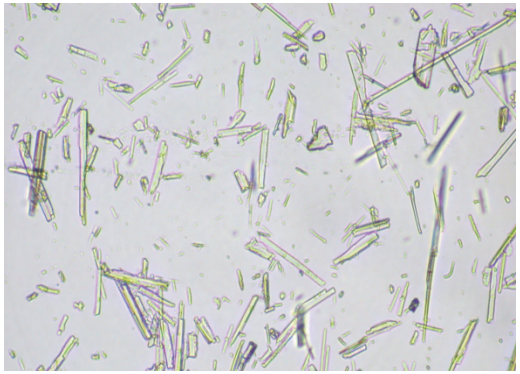
Particle Shape Analyzer

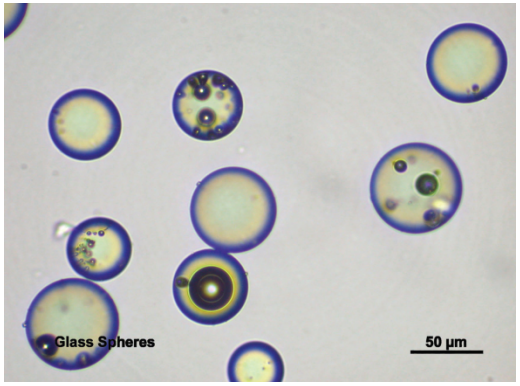
- **High-magnification optics**
- **High-resolution camera**
- **Uniform, controllable lighting necessary for accuracy and consistency**
- **Speed to achieve necessary particle count required for statistical validity in a reasonable time**
- **Several shape models to accommodate many types of particles**
- **Real time display and single image analysis mode, for high user confidence**
- **Can be configured for off-line or on-line mode.**

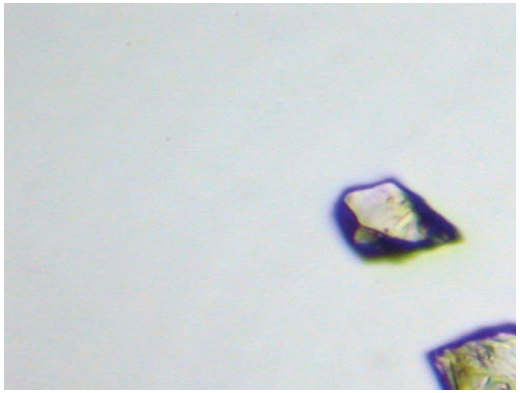
Particulate Systems

materials characterization WORKSHOP

Comparison of Particle Size Analysis Results







Particle Size Distribution Analysis

- Direct Measurement of Size
  - Microscopy and Image Analysis
  - Sieving, in a sense
- Indirect Measurement of Size
  - Measurement of Property that is a Function of Size
  - Equivalent Spherical Model
- Shape Dependent Interpretation

Particle Size Distribution Analysis

- Keys to Reliable Particle Size Analysis:
  - Proper sample selection
  - Proper sample preparation
  - Proper instrument operation
  - Proper analysis protocol
  - Proper report configuration
- Steps to ensure each

Sample Selection

- Proper Sample Division
  - Moving sample
  - Combine multiple small samples
  - Spinning riffler
  - May require multiple stages
- Sample Amount
  - Sufficient to give good statistics
  - Beware of particle to particle interactions
  - Appropriate to technique

Sample Preparation

- Sample Dispersion
  - Wetting
  - Dispersion
- Interparticle Forces
  - Aggregate
  - Agglomerate
  - Flocculate

Sample Dispersion

- Dispersion Stages
- Wetting the Sample
- Breaking Apart Agglomerates
- Stabilizing the Dispersion

General Dispersing Techniques

- Liquid Used
  - Ability to Pump or Suspend Largest Particles in Liquid Dispersions
  - Solubility of Particles
- Type of Surfactant Needed
  - Ionic
  - Nonionic
  - Wet and stabilize
- Amount of Surfactant
  - Amount of sample
  - Sample surface area, and thus, size

Critical Factors

- Agitation
- Shear Energy Level
  - Sufficient to deagglomerate
  - Appropriate to application
  - Friable samples
- Treatment Power versus Time

Critical Factors

- Agitation Equipment
- Increasing Power
  - Stirrer
  - Ultrasonic bath
  - Blender
  - Ultrasonic Probe
  - Probe with stirring
- More Power Requires Less Time for Same Dispersion

Dispersants

- Ionic
  - Polyphosphates - Calgon
  - Polyacrylates - Darvan
  - Polysulfonates - Daxad
  - Quarternary ammonium salts
- Non-ionic
  - Triton X-100

Water Precautions

- Dissolved Air
- Degassing During Analysis
  - Cavitation in fluid system
  - Cavitation by ultrasonic treatment
  - Temperature increase inside instrument
- Degassing Before Use
  - Heating
  - Evacuation
  - AquaPrep

Proper Instrument Operation

- Installed According To Manufacturers Specifications
- Initial Operation Verified
- Maintained on a Regular Basis
  - User Maintenance
  - Annual Preventive Maintenance
- Control Samples Analyzed Periodically

Proper Instrument Operation

- What To Do In Case Of Problems?
- Troubleshooting Guide
- Contact Manufacturers Representative
- Necessary Instrument Information
- History Leading up to Problem
- Diagnostic Testing
- Corrective Action

Proper Analysis Protocol

- Basically, how is the analysis to be performed?
- Protocols need to be specific for results to be reproducible.
- If a parameter can change the results then control it.
- Interlaboratory study to determine important factors, expected precision within and between labs.

Proper Analysis Protocol

- Each technique has its critical parameters.
- Consult Operator's Manual for guidance.
- Consult Experts in the field for additional help.
- Standardized within a company or an industry
- Look to published protocols for specific instructions.

Analysis Results

- Basis of the Analysis
  - Number
  - Area
  - Volume
  - Mass
- Frequency and Cumulative
  - Specific sizes
  - Cumulative finer or coarser

Analysis Results

- Summary Statistics
  - Mean, Median, Mode
  - Percentiles
  - Standard Deviation (width of distribution)
  - Other moments of the distribution
- Prediction of Behavior
  - Which statistics will best do this?
  - Research, Production, and Quality Control

Differences Between Techniques

- Volume versus Number Statistics
- Different Physical Phenomena
  - Sedimentation velocity – drag on envelope surface
  - Static light scattering – projected cross section, according to Mie Theory
  - Electrical Sensing Zone – particle volume
  - Dynamic Image Analysis – projected cross section
- Different Material Properties
  - Sedimentation – skeletal density
  - Static light scattering – refractive index
  - Electrical Sensing Zone – not based on specific properties
  - Dynamic Image Analysis – not based on specific properties
- Effect of shape
  - Definition of equivalent sphere

Differences Between Techniques

- Shape Effects upon Particle Size Distribution Calculations
- For two Samples, the one with larger particles as measured by one technique will usually be larger when measured by a different technique
- Correlation rather than Agreement
- Remember, reproducibility and prediction of behavior are desired

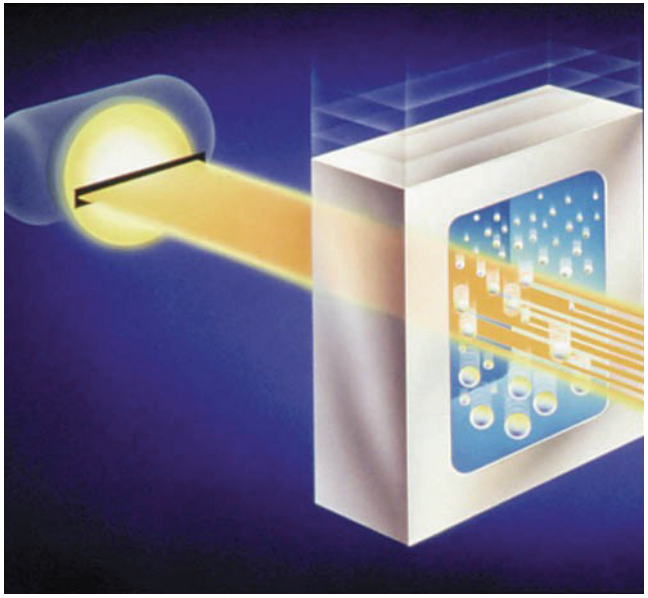
Differences Between Techniques

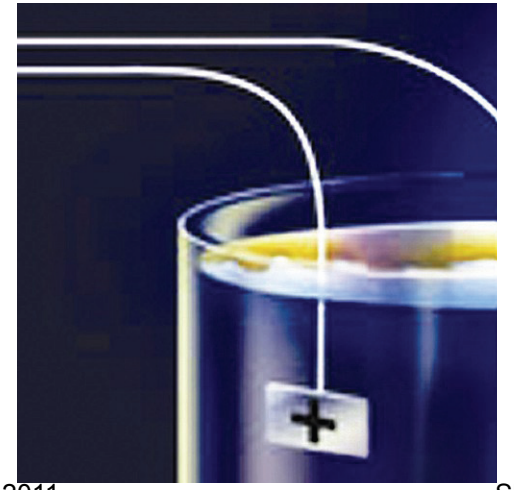
- Drag on Envelope Surface
- Ratio of Surface to Volume as a Function of Shape
- Scattering from Projected Area
- Orientation in Light Beam
- Orientation versus Polarization of Laser
- Assumption of lack particle influence on liquid viscosity and refraction
- Equivalent Spherical Diameter definition

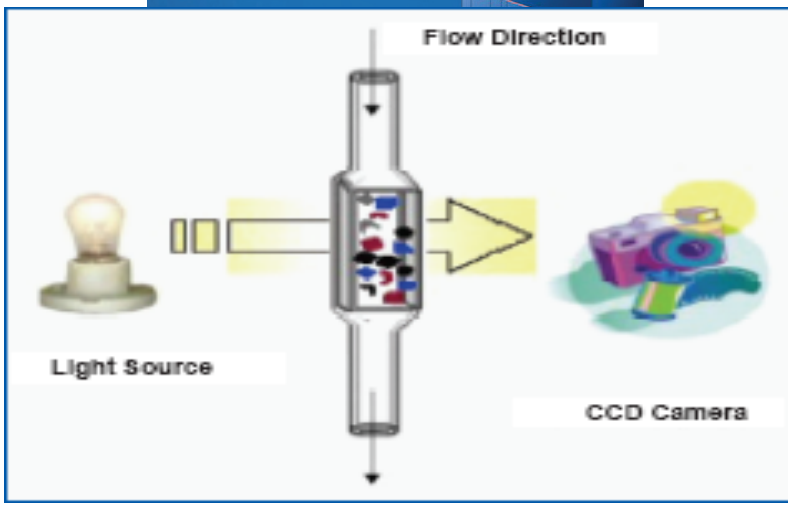
Differences Between Techniques

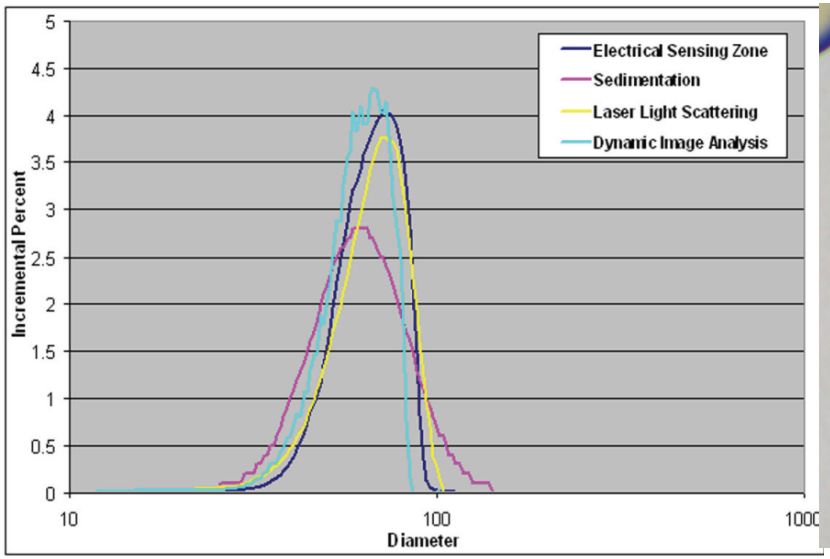
- Skeletal Density
- Closed Porosity
- Pores Filled with Dispersing Liquid
- Effective Density
- Refractive Index
- Complex, real and Imaginary
- Effects of Surface Roughness, Porosity
- Parameters of suspending fluid

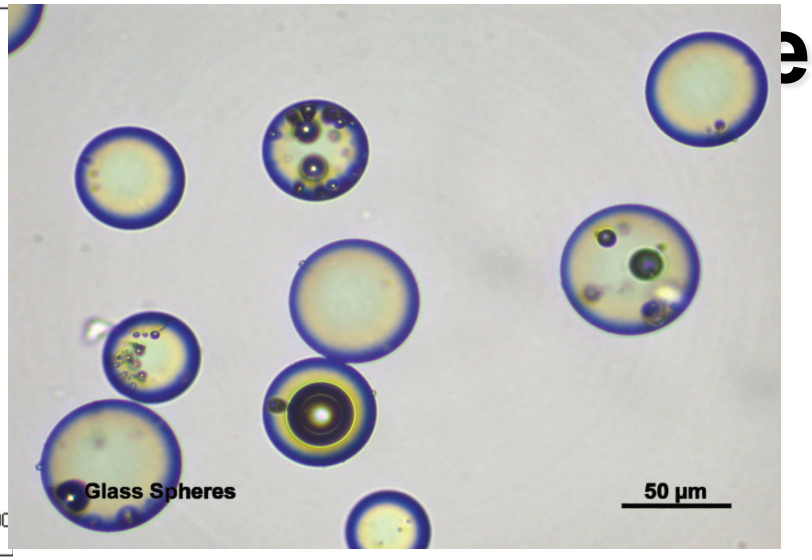
Differences Between Techniques





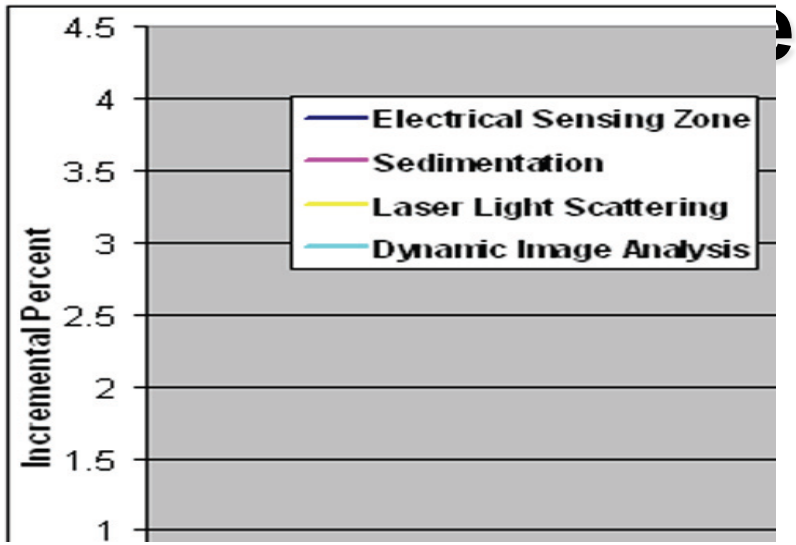


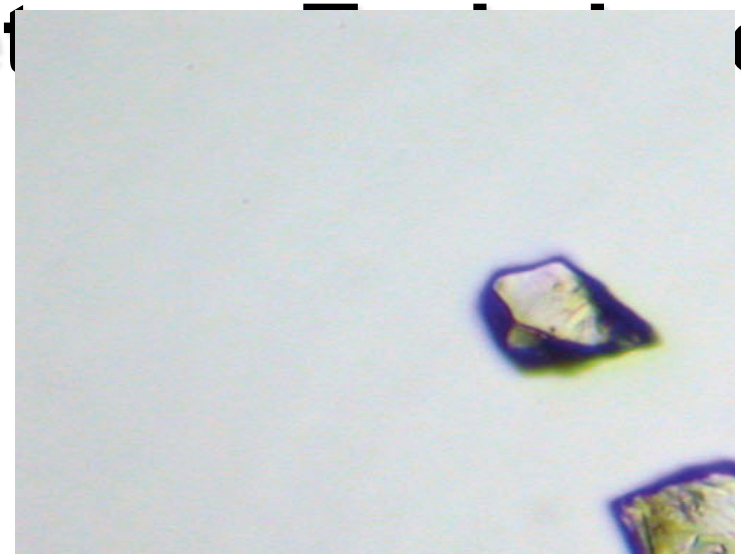




Technique	D10	D50	D90	Mode
Sedimentation	41.02	61.55	89.10	63.10
Laser Light Scattering	45.01	66.94	84.98	71.12
Electrical Sensing Zone	49.46	66.52	82.42	72.37
Dynamic Image Analysis	45.14	61.71	75.87	67.13

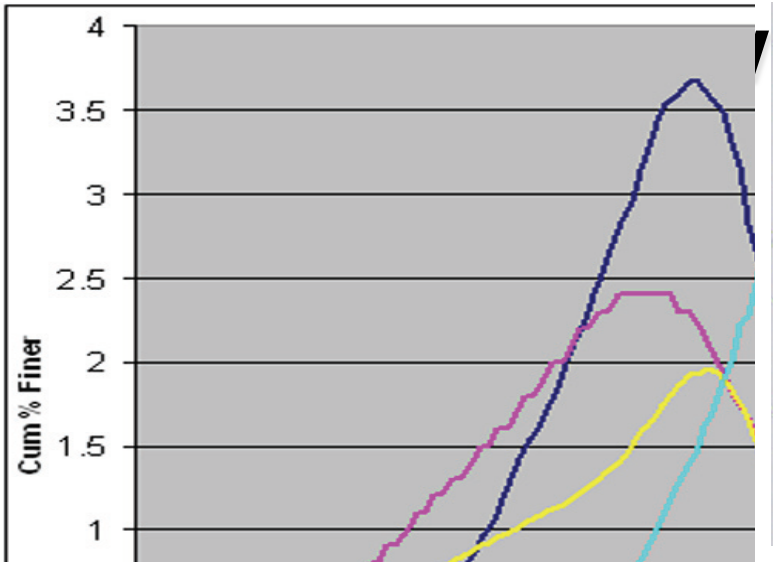
- Analyses of Glass Spheres using four particle size distribution techniques

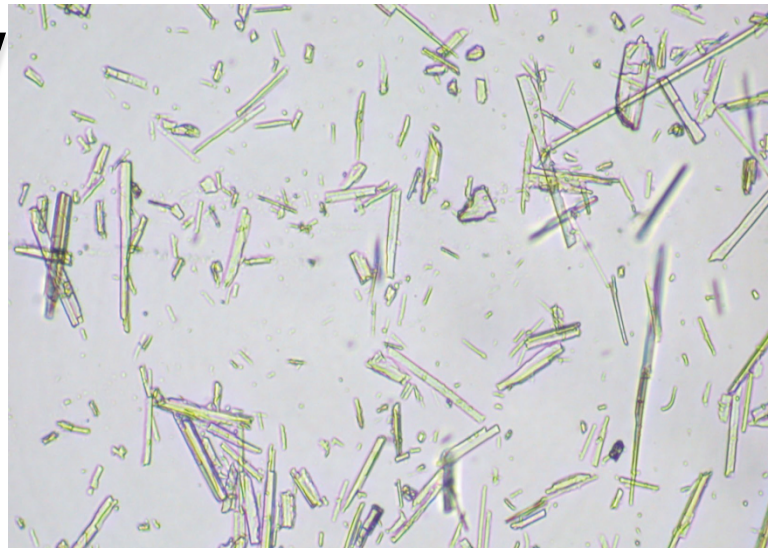




Technique	D10	D50	D90	Mode
Sedimentation	16.39	25.37	40.77	25.12
Laser Light Scattering	20.14	32.40	53.36	31.77
Electrical Sensing Zone	19.71	25.56	33.82	24.68
Dynamic Image Analysis	21.90	29.84	42.83	28.51

- Analyses of Garnet sample using four particle size distribution techniques





Technique	D10	D50	D90	Mode
Sedimentation	2.72	8.33	18.40	10.00
Laser Light Scattering	2.92	12.69	53.51	13.40
Electrical Sensing Zone	5.53	11.13	18.91	12.46
Dynamic Image Analysis	11.73	23.55	41.29	25.71

- Analyses of Wollastonite using four particle size distribution techniques

Particle Size Distribution Analysis

- So which is correct?
- Which one helps you predict behavior?
- Which measured property is closest to that which best describes the sample?
- What is Needed from Particle Size Distribution Analyses?
- Repeatability and Reproducibility
- Accuracy and Reliability
- Resolution and Sensitivity
- High Throughput

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Particle Size Distribution Analysis

- Keys to Reliable Particle Size Analysis:
  - Proper sample selection
  - Proper sample preparation
  - Proper instrument operation
  - Proper analysis protocol
  - Proper report configuration
- Steps to ensure each

Summary

- X-ray Gravity Sedimentation Particle Size Analysis
- Static Laser Light Scattering Particle Size Analysis
- Electrical Sensing Zone Particle Size Analysis
- Dynamic Light Scattering Particle Size Analysis
- Air Permeability Particle Size, and Surface Area, Analysis
- Dynamic Image Particle Shape Analysis

Thank You for Your Attention

Any Questions?