Particle size analyze

Asst. Prof. Dr. Aseel B. Al-Zubaidi

Methods for determining particle size

1. **Microscopy**
2. **Sieving**
3. **Laser light scattering techniques**
4. **Sedi mentation techniques**
5. **electrical sensing zone method**
6. **Surface area measurement techniques**

1. Microscopy

Optical microscopy (1-150µm)
Electron microscopy (0.001µ)

- **Being able to examine each particle alone has led to microscopy being considered as an absolute measurement of particle size.**
- **Can distinguish aggregates from single particles**
- **When coupled to** image analysis computers **each field can be examined, and a distribution obtained.**
- Number **distribution**
- **Most severe limitation of optical microscopy is the** depth of focus **being about 10µm at x100 and only 0.5µm at x1000.**
- **With small particles,** diffraction effects **increase causing spot at the edges - determination of particles < 3µm is less.**

For sub Micron particles it is necessary to use either

- TEM **(Transmission Electron Microscopy) or**
- SEM **(Scanning Electron Microscopy).**
- **TEM and SEM (0.001µm)**

Advantages

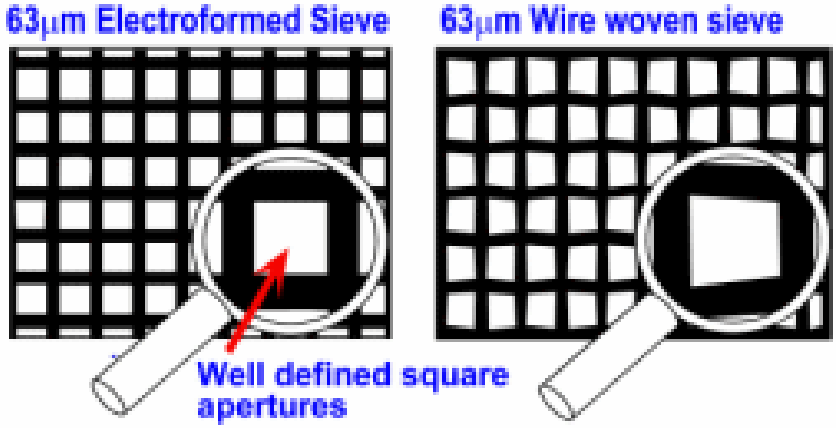
- **Relatively inexpensive**
- **Each particle individually examined - detect aggregates, 2D shape, colour, melting point etc.**
- **Permanent record - photograph**
- **Small sample sizes required**

Disadvantages

- **Time consuming - high operator fatigue - few particles examined**
- **Very low production**
- **No information on 3D shape**

2. Sieving

- Sieve analysis is performed using a nest or stack of sieves where each lower sieve has a smaller aperture size than that of the sieve above it.
- Sieves can be referred to either by their aperture size or by their mesh size (or sieve number).
- The mesh size (is the number of wires per linear inch).
- Approx. size range : 5µm - ~3mm
  - ✓ Standard woven wire sieves
  - ✓ Electroformed micromesh sieves at the lower end or range (< 20µm)
  - ✓ Blow plate sieves at the upper range.



- **Sieving may be performed** wet **or** dry**; by** machine **or by** hand**, for a** fixed time **or until powder passes through the sieve at a** constant low rate
- **Wet sieving**
- **Air-jet sieving**
- Weight **distribution**

Advantages

- **Easy to complete**
- **Wide size range**
- **Inexpensive**

Disadvantages

- **Wear/damage in use or cleaning**
- **Rough particles**
- **Rod-like particles : overestimate of under-size**
- **Work hard**

3.Laser Light Scattering

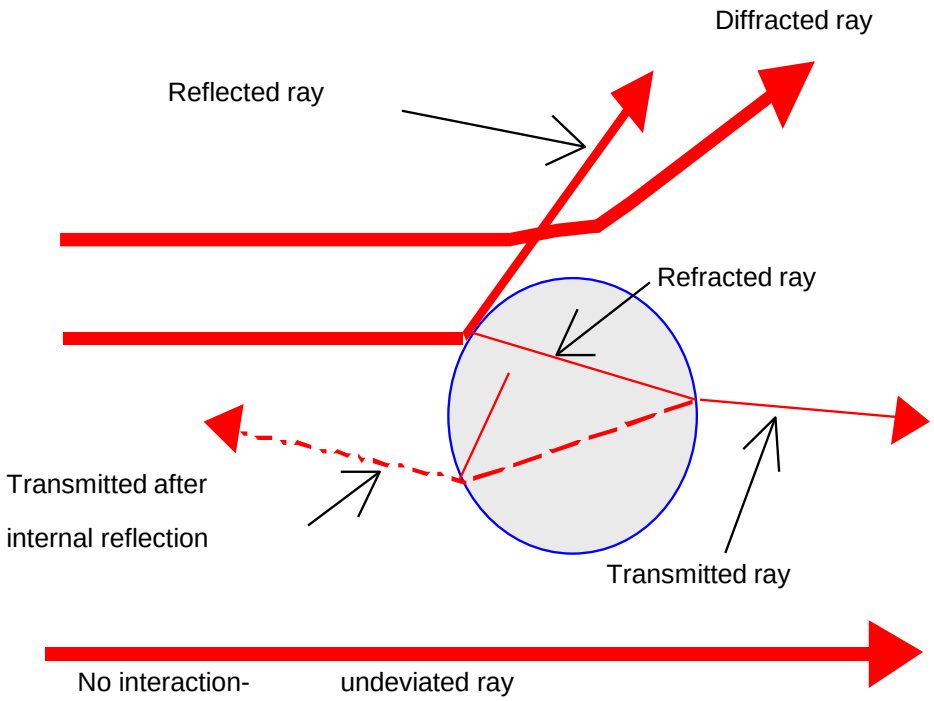
1. **Widely used fast technique that can be applied to various particulate systems**
2. **Easily automated in a variety of commercially available instruments**
3. **Requires knowledge of Refractive Index of the material (n)**
4. **Data presented as Equivalent Spherical Diameter**

Historically *laser scattering (angles)*

- **was performed at small angles only, typically up to 14 ˚, called**
  - ✓ **Fraunhofer diffraction**
  - ✓ **Forward light scattering**
  - ✓ **Low-angle laser light scattering (LALLS)**
- **Gave results down to** 1 µm
- **Now broadened to include wider angles with Mie Theory optical modeling**
- **Expanded range down to** 0.1 µm

Types of Scattered Light

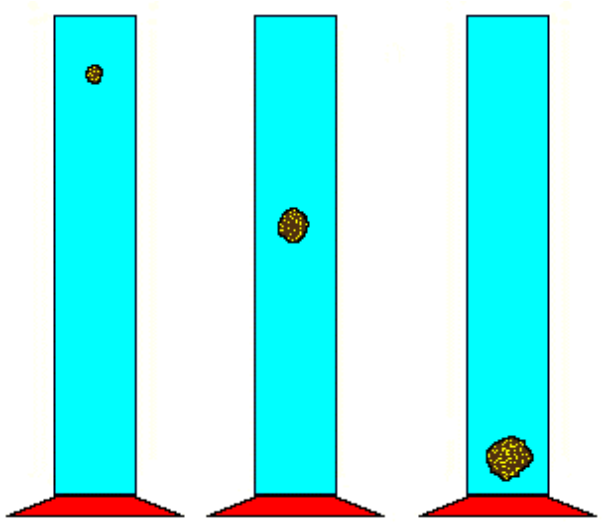
- Diffraction
- Reflection
- Refraction
- Absorption

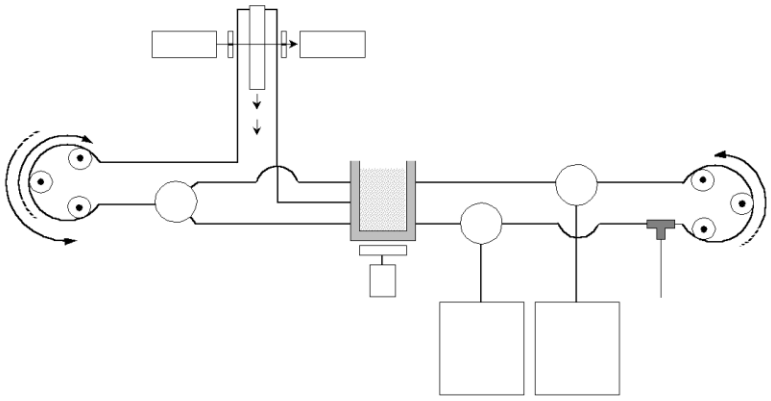


4.X-ray Se dimentation

- Based Upon a Classical Particle Sizing Method - Stokes' Law.
- Same Sizing Principle as Andreasen Pipette and Long-Arm Centrifuge.
- Direct Mass Concentration Detection.
- X-Ray Attenuation is Proportional to Mass in Beam.
- Material Density must be Known
- Widely used in the Ceramic Industry

- Stoke's Law for Sedimentation, 1891.
- Mature, well understood and widely practiced technique.
- Now fully automated





Stoke's equation

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

D = particle diameter

η = liquid viscosity

v = sedimentation velocity

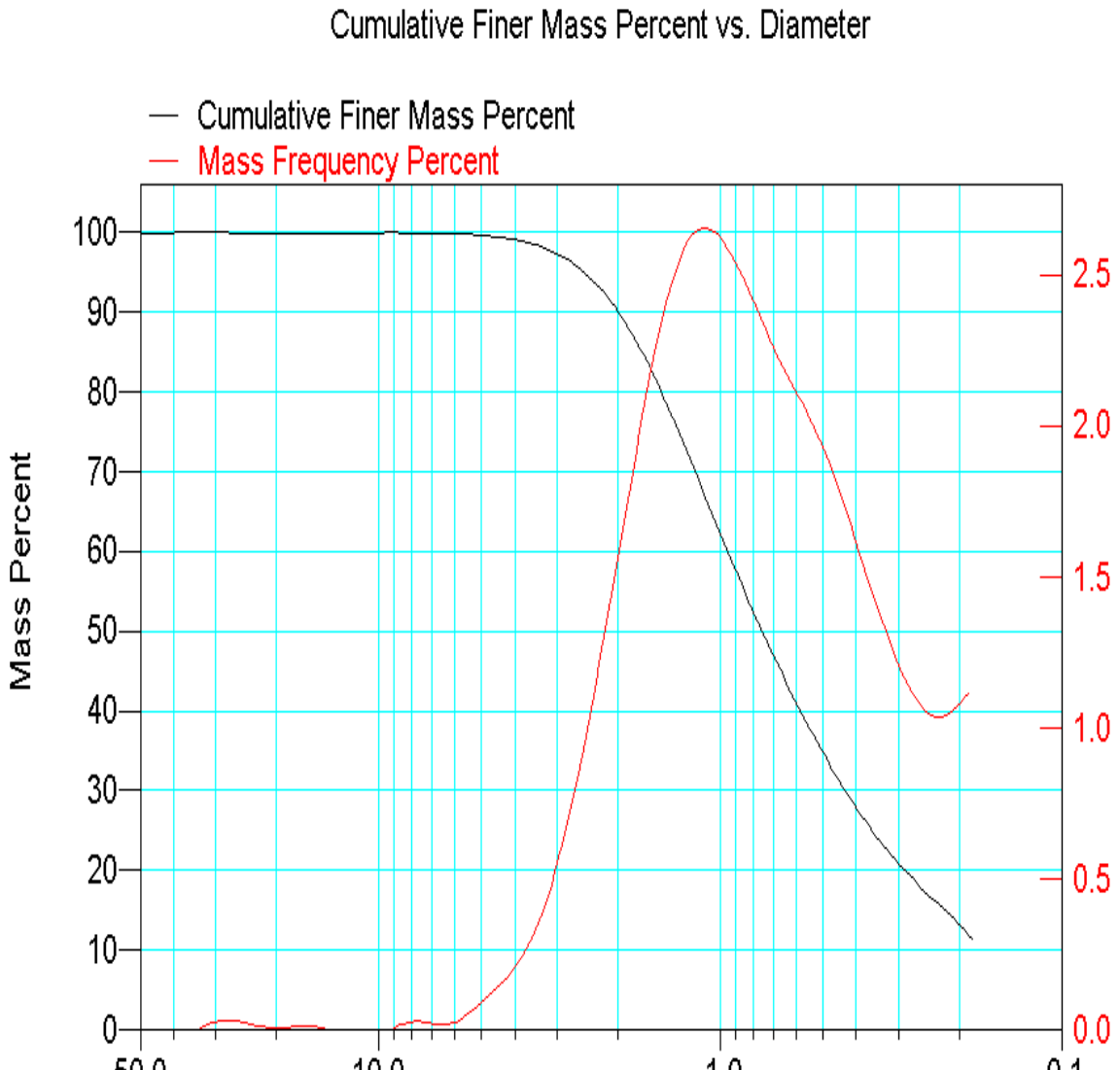
ρ = particle density

ρ_o = liquid density

g = acceleration due to gravity

- Accounts for Particle Mass Outside Analysis Range.
- Analyzes Higher Concentrated Slurries than Most Other Techniques.
- Provides Reliable Analyses of Wide Size Range: 300 µm to 0.1 µm.
- Requires Only Readily-Available Physical Constants as Parameters.

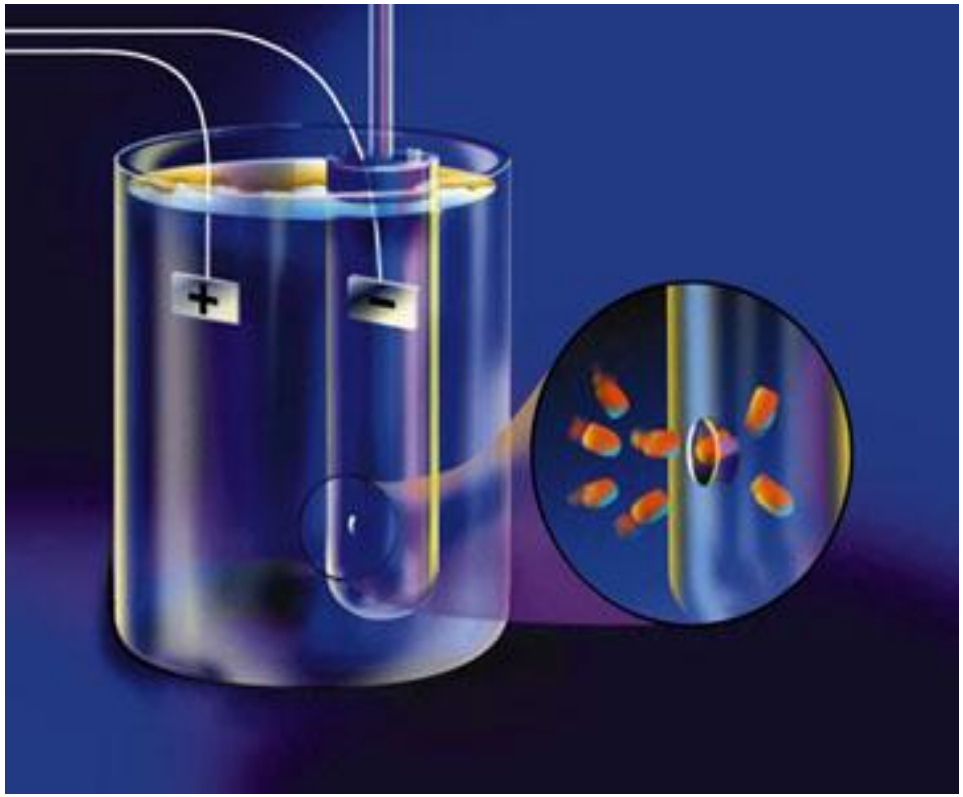
Particle Size Distribution Analysis of Fine caco3 Sample.



5.Electrical Sensing Zone

- **Particle suspended in a conductive liquid, passing through a narrow orifice, increases resistance through the orifice**
- **Voltage must increase to keep constant current through orifice, same as Elzone 5380 and Multisizer 3**
- **Voltage change proportional to particle volume**

Elzone Principle of Operation



Advantages

1. Counts and sizes organic and inorganic particles
2. Analyzes materials with mixed optical properties, densities and shapes
3. Higher resolution than other sizing methods
4. Lower quantity of sample needed for accurate, easy analysis
5. Compact size takes up little lab space
6. Automatic orifice blockage detection

6. (Surface area measurement techniques)

- **Increasing interest in nanomaterials**
  - ✓ **Particle sizes < 100 nm are of interest**
  - ✓ **Most techniques in this range questionable**
    - **Dynamic Light Scattering**
      - **Provides Mean Size**
      - **Difficult if bimodal**
  - ✓ **Agglomerates make sizing difficult**
- **Obtain average size of primary particles from the surface area and density of the material**

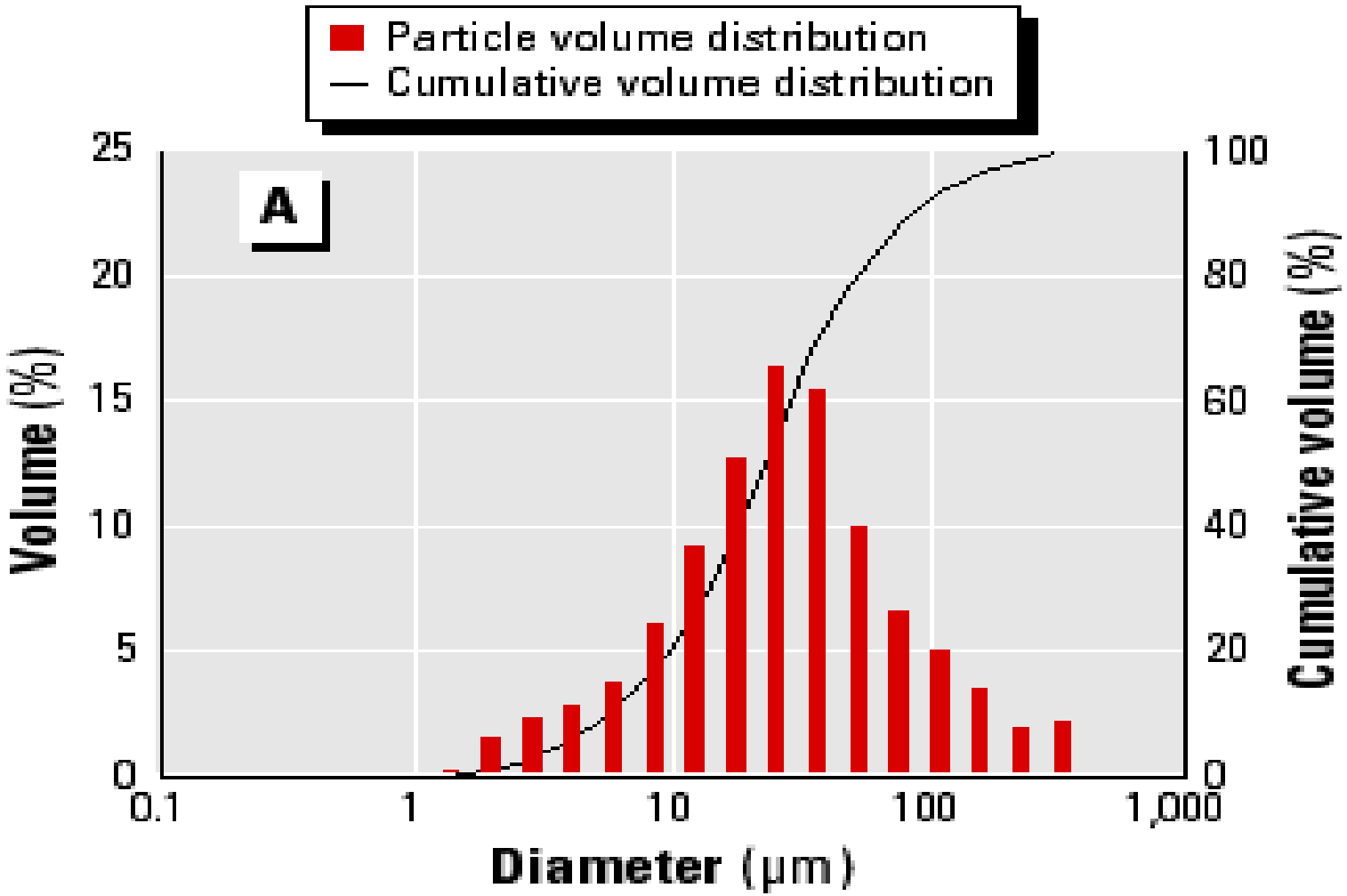
Particle Size from Surface Area

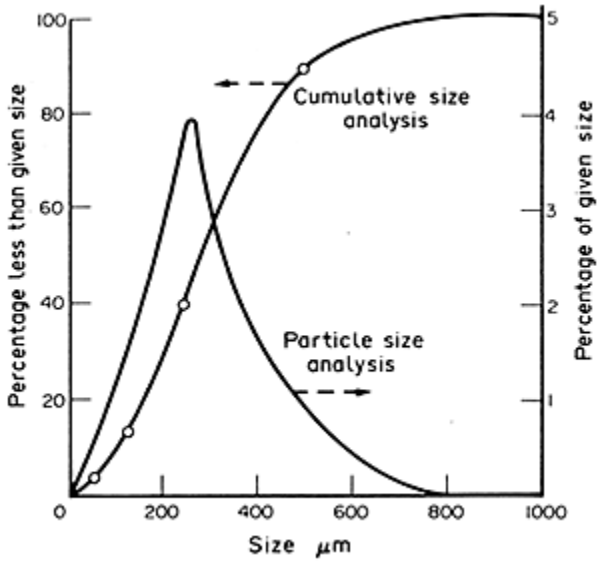
$$\frac{1}{\text{BET} \times \text{Density}} = \frac{\text{Volume } (4/3)\pi r^3)}{\text{Area } (4\pi r^2)}$$

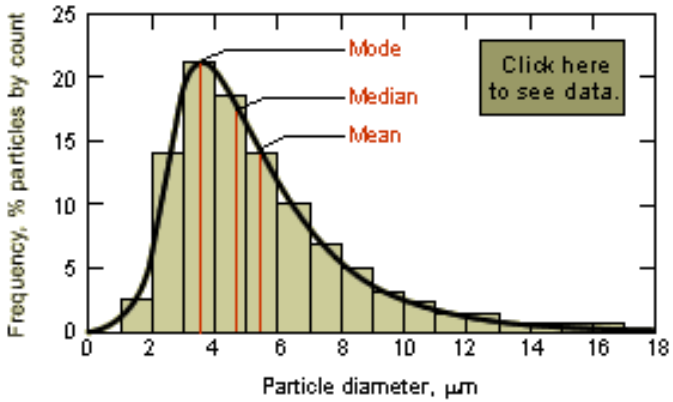
(Brunauer, Emmett and Teller)

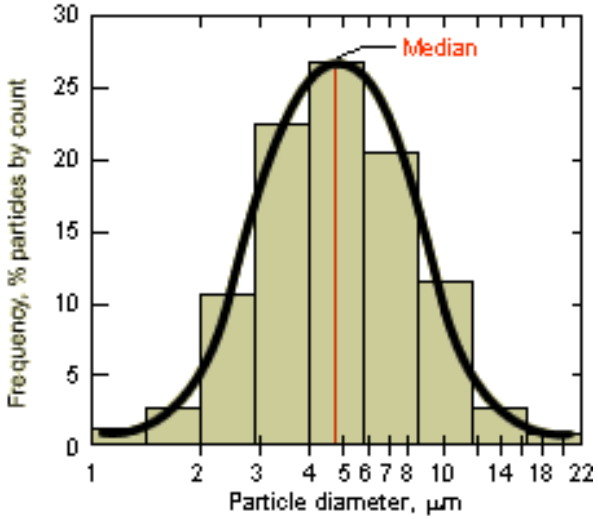
Reduces to:

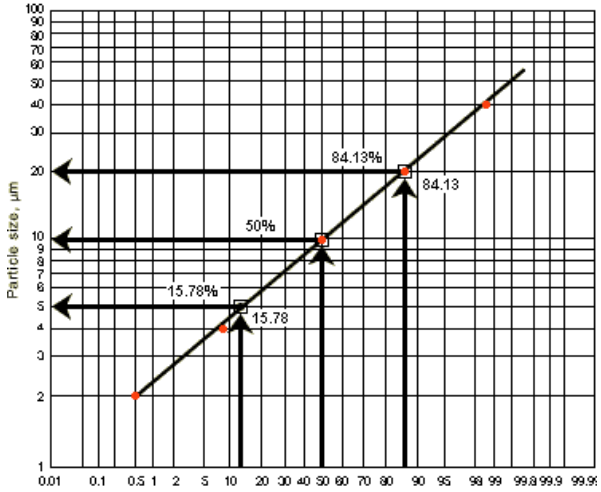
$$D\,(\text{nm}) = \frac{6}{\text{BET} \times \text{Density}} \times 1000$$


Particle volume distribution
Cumulative volume distribution
A
Volume (%)
Cumulative volume (%)
Diameter (µm)
0
5
10
15
20
25
0
20
40
60
80
100
0.1
1
10
100
1,000

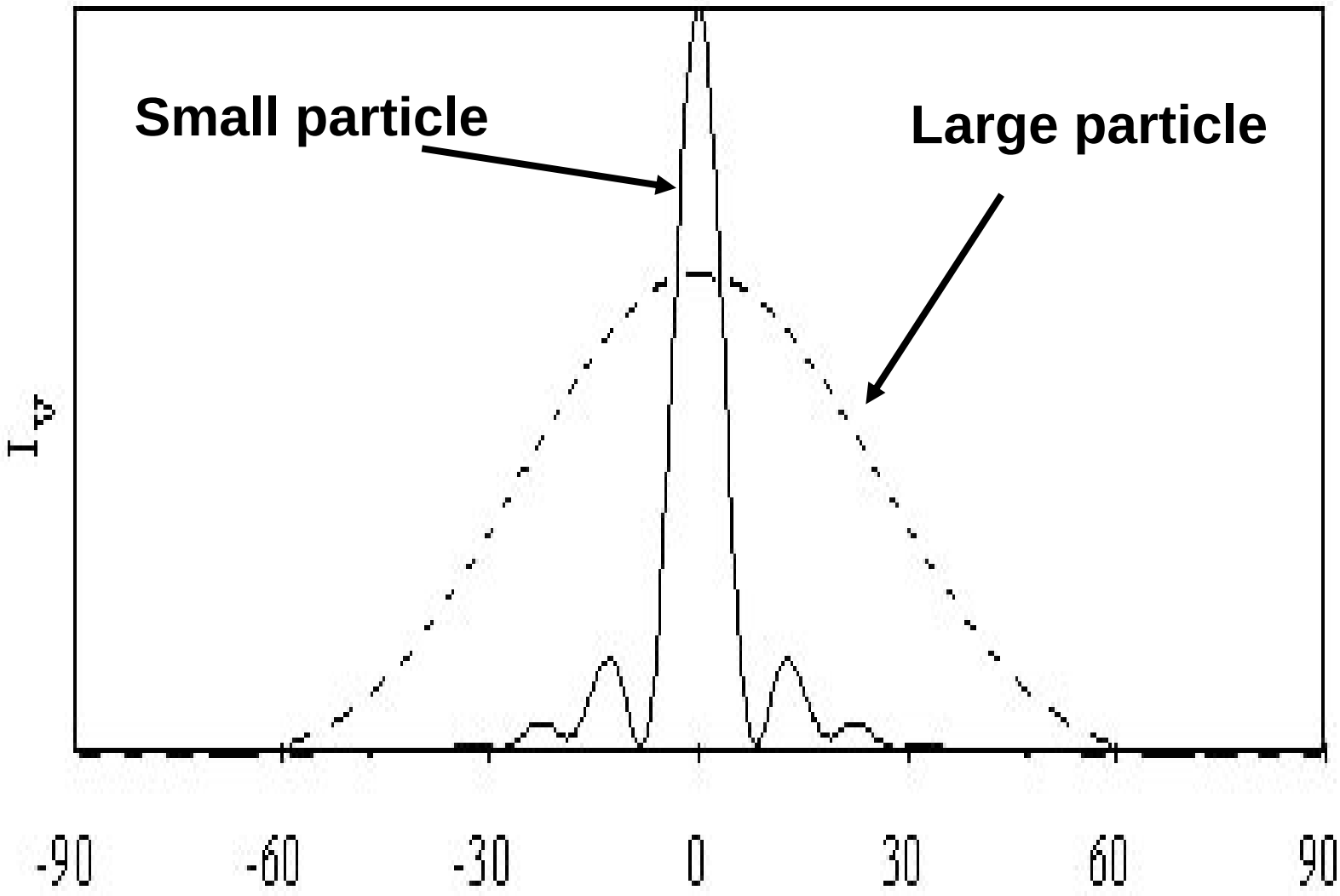

Percentage less than given size
Percentage of given size
Cumulative size analysis
Particle size analysis
Size μm
0
200
400
600
800
1000
20
40
60
80
100
1
2
3
4
5

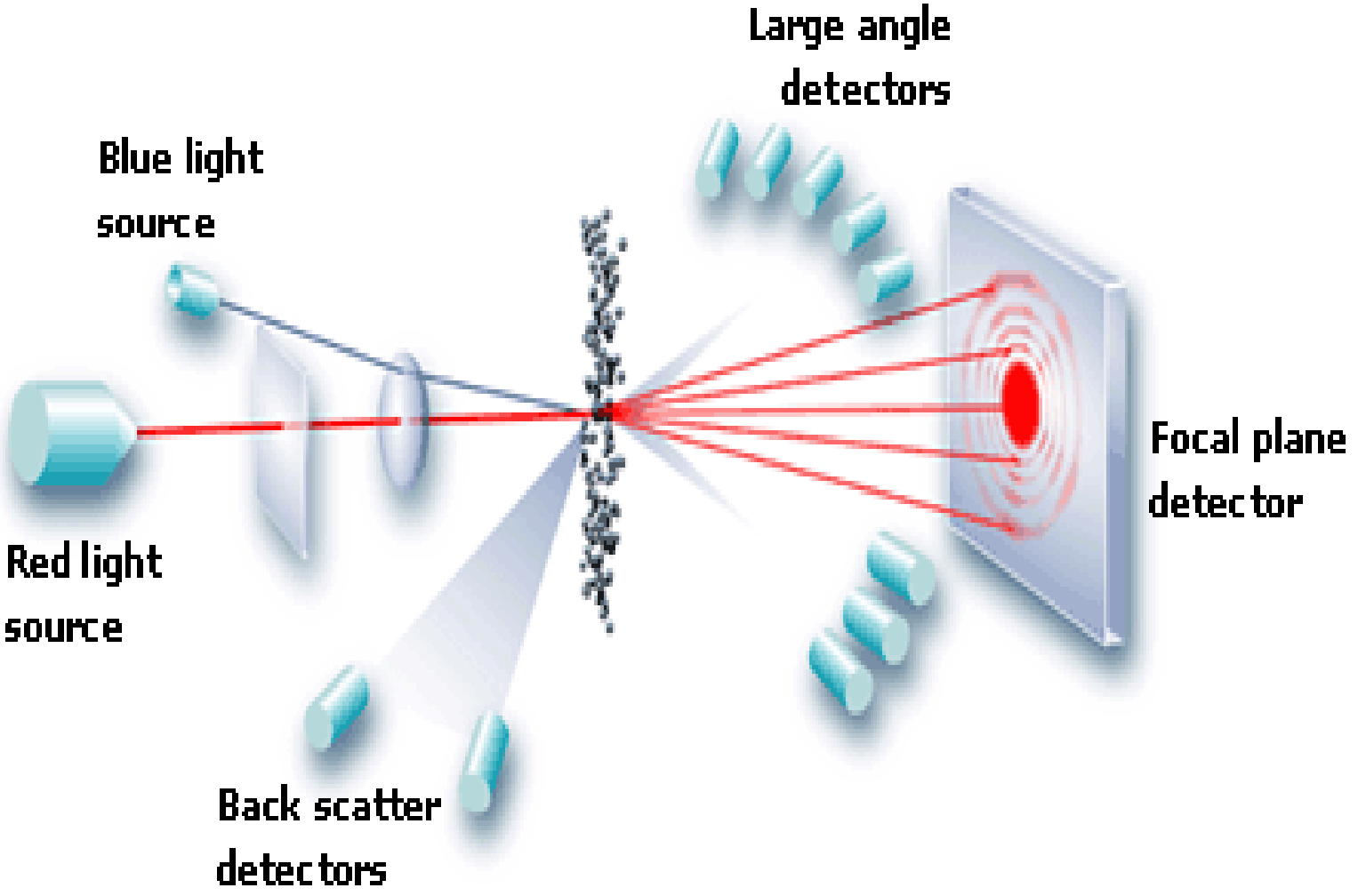

Frequency, % particles by count
Particle diameter, μm
Mode
Median
Mean
Click here to see data.
0
2
4
6
8
10
12
14
16
18
5
10
15
20
25


Frequency, % particles by count
Particle diameter, μm
Median
1
2
3
4
5
6
7
8
10
14
18
22
0
5
10
15
20
25
30

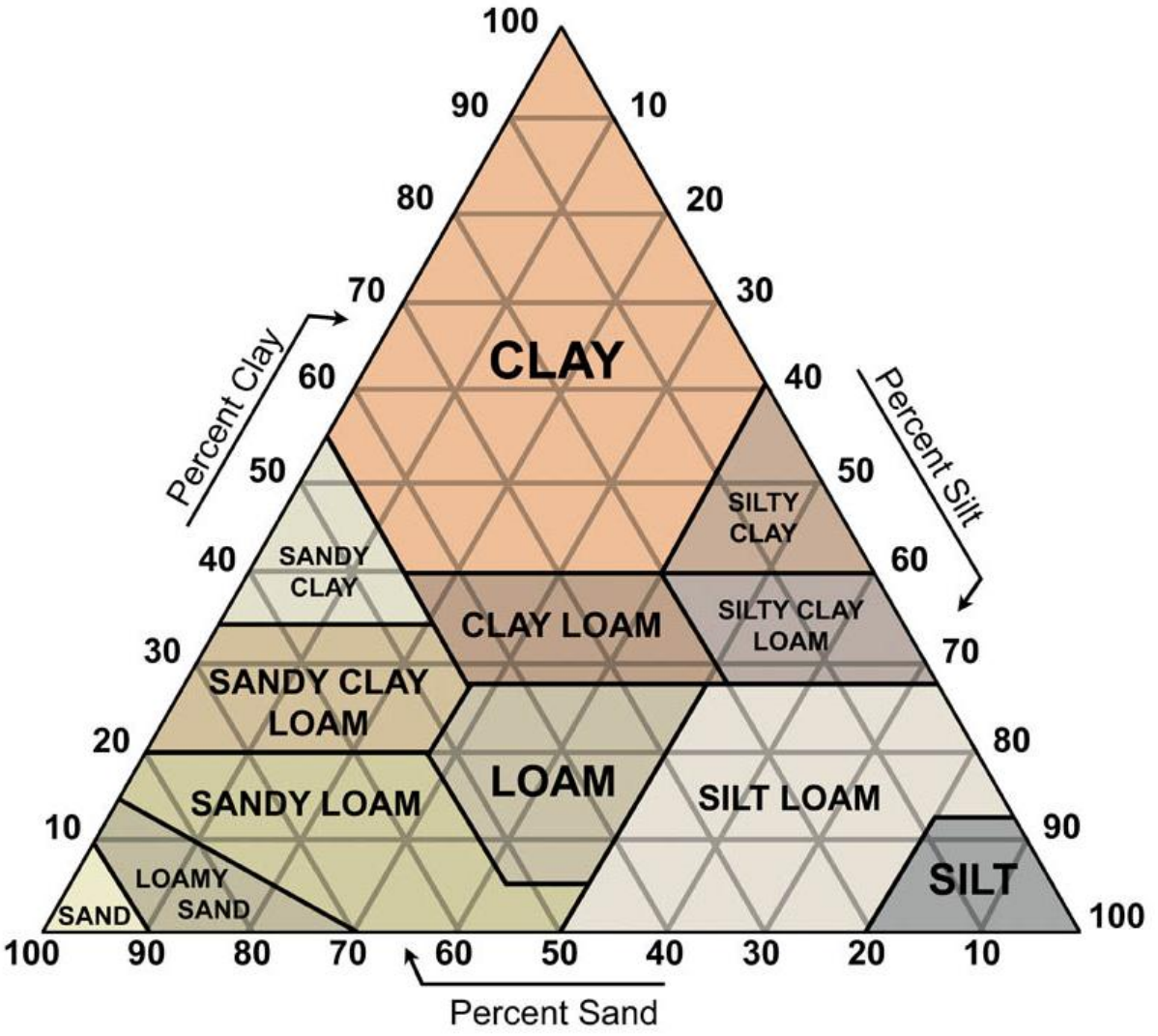

Particle size, μm
84.13%
84.13
50%
15.78%
15.78

Measuring : Laser diffraction




Large angle
detectors
Blue light
source
Red light
source
Focal plane
detector
Back scatter
detectors

Quick and easy, but not really a distribution



Particle Size Techniques

- **Laser Light Scattering**

- **X-ray Sedimentation**

- **Electrical Sensing Zone**