

## Determination of Particle Size Distributions Using Laser Diffraction

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### Introduction

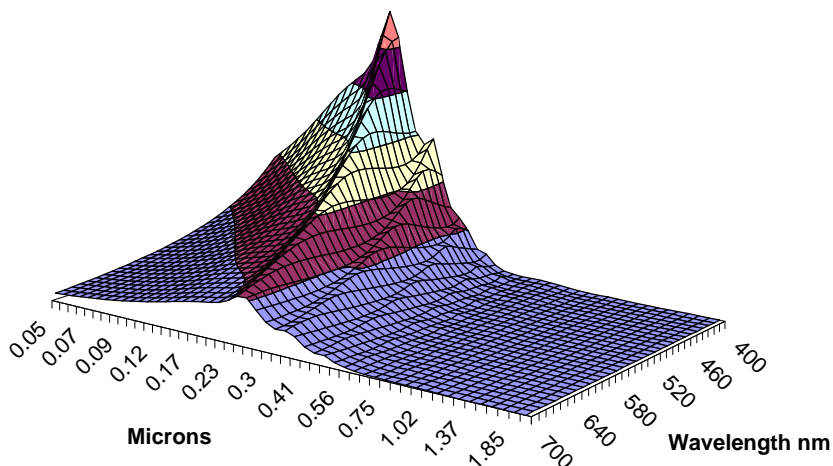
In recent years particle sizing using the laser diffraction techniques has become the dominant method of choice. Laser diffraction units do not measure particle size distributions but carry out light scattering experiments. The relationship between the light scattered by the particles and the final particle size distribution reported depends critically upon assumptions made about the optical properties of the material under study. While the previous sentences are statements of the obvious, misleading results continue to be published due to the potential influence of time pressure, psychology and lack of formal training in optics.

### Dependence of light scattering on particle size and refractive index

Light scattering occurs at refractive index gradients. If transparent glass spheres of refractive index  $1.498 - 0.0$  are dropped into a beaker of clean benzene (refractive index  $1.498 - 0.0$ ) they will no longer be visible. This is a condition of index matching. The higher the refractive index of the particle relative to the medium in which it is suspended, the more light will be scattered. Titanium dioxide ( $\text{TiO}_2$ ) makes a strong white pigment because it has a high refractive index. The Anatase form has a refractive index of  $2.54 - 0.0$ . Note: The first number in the refractive index number-pair  $1.498 - 0.0$  is the real part of the refractive index ( $n$ ) and the second is the imaginary part ( $k$ ), where the complex refractive index is defined as  $n^* = n(1 - ik)$ . If the second number is zero the material is transparent..

For homogeneous spherical particles much larger than the wavelength of the light, the scattering power varies increases in proportion to their geometric cross section or diameter squared, ( $D^2$ ) the scattered light is also concentrated at low angles. For spherical particles very much smaller than the wavelength of the light the scattering power decreases in proportional to  $D^6$  and the scattering is isotropic. When the wavelength is similar to the particle size the scattering power is a complicated function of particle size. It has a pronounced peak due to a resonant relationship between the size of the particle, its refractive index and the wavelength of the illumination (see Fig 1.) and the scattered light extends to high angles.

Fig. 1



**Fig. 1** -- Scattering efficiencies determined at zero angle, for equal volumes of anatase spheres  $RI = 2.54$ , plotted against particle size and wavelength in the visible wavelength region.

Pigment manufacturers try to take full advantage of the peak in the scattering power. For anatase the optimum particle size is about 0.25 microns. Figure 1 takes no account of the spectral response of the eye or of the spectral power of the illuminating source. Both of these latter points condition our appreciation of colour. Anatase pigment is nearly white because it adsorbs very little but scatters all the visible wavelengths (400 to 700 nm) nearly equally well. A yellow pigment would absorb the blue end of the visible spectrum very strongly.

With our present understanding of the physics of light scattering we can analyze the pattern of light scattered from a sphere as a function of wavelength and determine what size particle would cause this pattern.

### Dependence of light scattering on the number of particles in the beam

The amount of light scattering signal recorded at the detector is a function of the scattering power of the particles. The scattering power depends on the refractive index of the solid relative to the surrounding medium. The refractive index is a complex spectral function with both real and imaginary parts. The scattering power also depends on the particle's scattering cross-section and (in the case of a distribution) the volume (or number) of particles of each size. At this point one should remember that a single 1000  $\mu\text{m}$  spherical particle has the same volume as a million 10  $\mu\text{m}$  spherical particles and that a single 1000  $\mu\text{m}$  spherical particle has the same light-scattering power as ten thousand 10  $\mu\text{m}$  particles.

At low concentrations (single scattering), doubling the concentration of particles of any one size will double the scattering contribution made by that size fraction to the total light scattering seen by the detectors. Laser diffraction instruments usually report the volume (rather than the number) of particles that have a given size because the light scattering signal is proportional to

D<sup>2</sup>. Discriminating the tiny scattering pattern from a single small particle in the presence of the huge scattering signal from a single large particle is very difficult. If enough small particles are present to equal the volume of the single large particle the scattering of the small particles can be discriminated (separated out) and analyzed. This is why laser diffraction units usually report the volume of particles in each size class.

### **Dependence of light scattering on the angle of observation**

Calculations using Mie theory (Ref. 1) require an input of the following parameters:

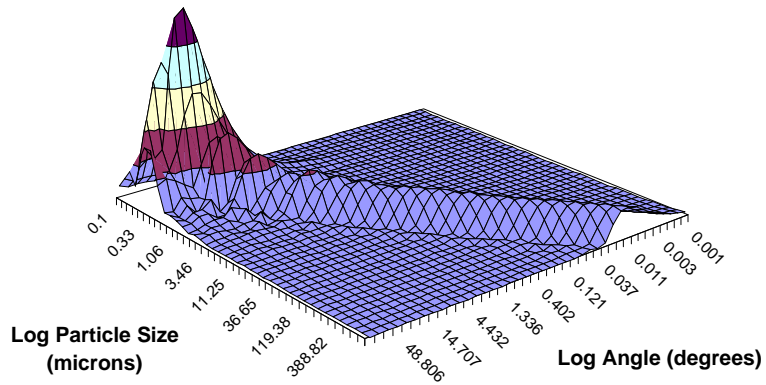
- wavelength of illumination
- the polarisation state of the illumination
- the refractive index -- both real and imaginary -- of both the particle and the medium
- the diameter of the particle
- the angle of observation relative to the incident illumination (In both azimuthal planes).

The output value returned is the intensity of the scattered light from a single spherical particle viewed at the selected angle from the incident illumination. For each diameter the scattered intensity may be divided by the cube of the diameter to give the scattering intensity per unit volumes of particles for that diameter.

A particle-size instrument based on light scattering can distinguish the scattering patterns of large particles from small particles because large particles scatter strongly and principally to small angles away from the incident light beam while small particles scatter weakly and too much larger angles. Analysis of the scattering intensity as a function of observation angle can yield a particle size distribution. Rather than using a single photo-detector to scan through many angles most instrument manufacturers use an array of photo-detectors at fixed positions. It is helpful if the photo-detectors provide equal values of output current, at their angular positions for equal volumes of particles in each size class. Since a photo-detector provides an output current proportional to the intensity of the illumination multiplied by the area of the detector, laser diffraction instrument designers use small-area detectors close to the incident laser beam (low angles) and detectors with progressively larger area at larger angles of observation. The amount of light scattered into the tiny detectors (at small angles) is most influenced by the large particles, which scatter the light intensity to small angles. The amount of light scattered into the larger area detectors (at higher angles) is most influenced by the small particles, which scatter light up to the higher angles. By this means laser diffraction designers effect a balancing process which aims to provide equal information about all sizes of particles. The current output from the array of detectors at many angles is analyzed in a complex and proprietary method to arrive at a particle volume distribution.

Figure 2 illustrates the angular dependence of scattered energy using a simple weighting factor for instrument detector geometry to illustrate an ideal situation.

Fig. 2



**Fig 2.** Light scattering energy (calculated using Mie theory) as a function of particle size and observation angle. The example is for anatase (RI = 2.54 – 0.0) in water (RI = 1.33) illuminated with He-Ne laser light (wavelength 633 nm). The particle sizes are from 0.1-microns to 1000-microns over angles ranging from 0.001-degrees to 120-degrees. The example uses equal volumes of particles for all sizes and was weighted to illustrate theoretically optimum detector geometry.