THE LA-950 LASER DIFFRACTION TECHNIQUE

The LA-950 combines the most popular modern sizing technique with state of the art refinements to measure wet and dry samples measuring 10 nanometers to 3 millimeters. The central idea in laser diffraction is that a particle will scatter light at an angle determined by that particle's size. Larger particles will scatter at small angles and smaller particles scatter at wide angles. A collection of particles will produce a pattern of scattered light defined by intensity and angle that can be transformed into a particle size distribution result. This technical note explains the basic underlying principles used by the LA-950 particle size analyzer.

Introduction

The knowledge that particles scatter light is not new. Rayleigh scattering of light from particles in the atmosphere is what gives the sky a blue color and makes sunsets yellow, orange, and red. Light interacts with particles in any of four ways: diffraction, reflection, absorption, and refraction. Figure 1 shows the idealized edge diffraction of an incident plane wave on a spherical particle. Scientists discovered more than a century ago that light scattered differently off of differently sized objects. Only the relatively recent past, however, has seen the science of particle size analysis embrace light scattering as not only a viable technique, but the backbone of modern sizing.

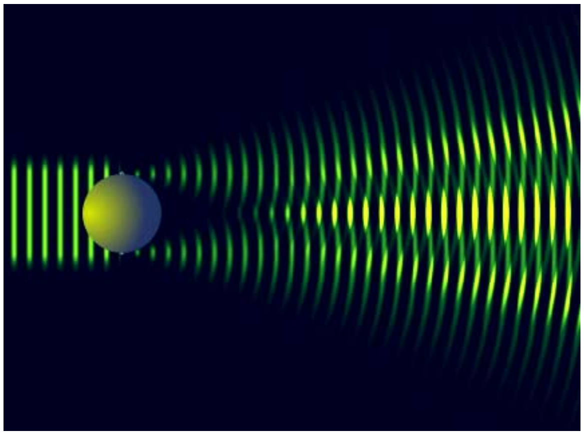

Figure 1: The diffraction pattern (right side) of a plane wave scattering off a spheroid (left side).

Bench-top laser diffraction instruments became practical with the advent of high intensity, reasonably priced lasers and sufficient computing power to process the scattered light data. Once these barriers to market entry were eliminated the advantages of laser diffraction over other techniques were apparent: speed of analysis, application flexibility, small particle accuracy, and ease of use. The ability to measure nano, micro and macro-sized powders, suspensions, and emulsions, and to do it within one minute, explains how laser diffraction displaced popular techniques such as sieving, sedimentation, and manual microscopy.

Such an instrument consists of at least one source of high intensity, monochromatic light, a sample handling system to control the interaction of particles and incident light, and an array of high quality photodiodes to detect the scattered light over a wide range of angles. This last piece is the primary function of a laser diffraction instrument: to record angle and intensity of scattered light. This information is then input into an algorithm which, while complex, reduces to the following basic truth:

> **LARGE PARTICLES SCATTER INTENSELY AT NARROW ANGLES**
>
> **SMALL PARTICLES SCATTER WEAKLY AT WIDE ANGLES**

The algorithm, at its core, consists of an optical model with the mathematical transformations necessary to get particle size data from scattered light. However, not all optical models were created equally.

The Importance of Optical Model

In the beginning there was the *Fraunhofer Approximation* and it was good. This model, which was popular in older laser diffraction instruments, makes certain assumptions (hence the approximation) to simplify the calculation. Particles are assumed…

- to be spherical
- to be opaque
- to scatter equivalently at wide angles as narrow angles
- to interact with light in a different manner than the medium

Practically, these restrictions render the Fraunhofer Approximation a very poor choice for particle size analysis as measurement accuracy below roughly 20 microns is compromised.

The *Mie scattering theory* overcomes these limitations. Gustav Mie developed a closed form solution (not approximation) to Maxwell's electromagnetic equations for scattering from spheres; this solution exceeds Fraunhofer to include sensitivity to smaller sizes (wide angle scatter), a wide range of opacity (i.e. light absorption), and the user need only provide the refractive index of particle and dispersing medium. Accounting for the light that refracts through the particle (a.k.a. secondary scatter) allows for accurate measurement even in cases of significant transparency. The Mie theory likewise makes certain assumptions that the particle…

- is spherical
- ensemble is homogeneous
- refractive index of particle and surrounding medium is known

Figure 2 shows a graphical representation of Fraunhofer and Mie models using scattering intensity, scattering angle, and particle size[1]. The two models begin to diverge around 20 microns and these differences become pronounced below 10 microns. Put simply, the Fraunhofer Approximation contributes a magnitude of error for micronized particles that is typically unacceptable to the user. A measurement of spherical glass beads is shown in Figure 3 and calculated using the Mie (red) and Fraunhofer (blue) models. The Mie result meets the material specification while the Fraunhofer result fails the specification and splits the peak. The over-reporting of small particles (where Fraunhofer error is significant) is a typical comparison result.

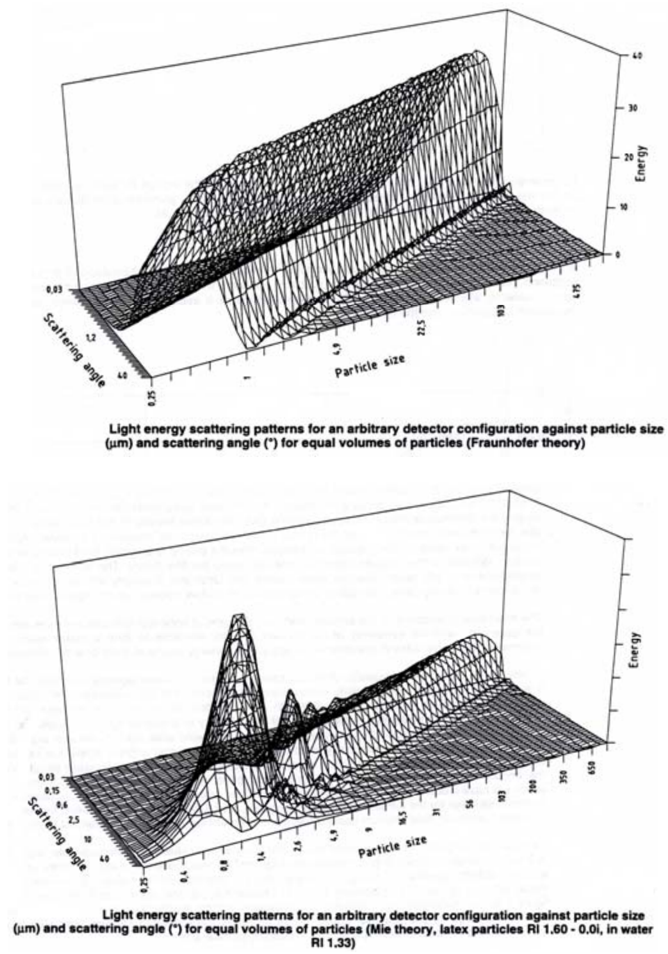

Figure 2: Representations of Fraunhofer (top) and Mie (bottom) scattering models using angle, energy, and size as parameters.

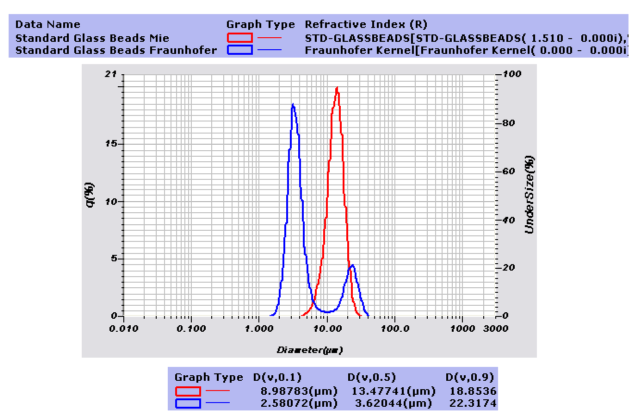

Figure 3: Mie (red) and Fraunhofer (blue) results for spherical glass beads

Building a State of the Art Laser Diffraction Analyzer

The basics of what needs to be measured and how it's transformed into particle size data are understood[2]. What constitutes a basic particle size analyzer has also been discussed, but there's a wide gulf between bare minimum and state of the art. The latter is always the industry leader in accuracy, repeatability, usability, flexibility, and reliability. The current state of the art in laser diffraction is the *Partica* LA-950 featuring two high intensity light sources, a single, continuous cast aluminum optical bench (Figure 4), a wide array of sample handling systems, and expert refinements expected from the fifth revision in the 900 series.

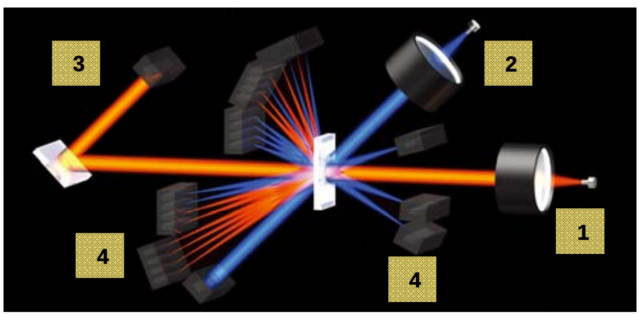

Figure 4: A simplified layout of the LA-950 optical bench. 1: Red wavelength laser diode for particles > 500 nm, 2: Blue LED for particles < 500 nm, 3: Low angle detectors for large particles, 4: Side and back angle detectors for smaller particles.

Using two light sources of different wavelengths is of critical importance because the measurement accuracy of small particles is wavelength dependent. Figure 5 shows the 360° light scattering patterns from 50 nm and 70 nm particles as generated from a 650 nm red laser. The patterns are practically identical across all angles and the algorithm will not be able to accurately calculate the different particle sizes. Figure 6 shows the same experiment using a 405 nm blue LED. Distinct differences are now seen on wide angle detectors which allows for accurate calculation of these materials. Integrating a second, shorter wavelength light source is the primary means of improving nano-scale performance beyond the bare minimum laser diffraction analyzer.

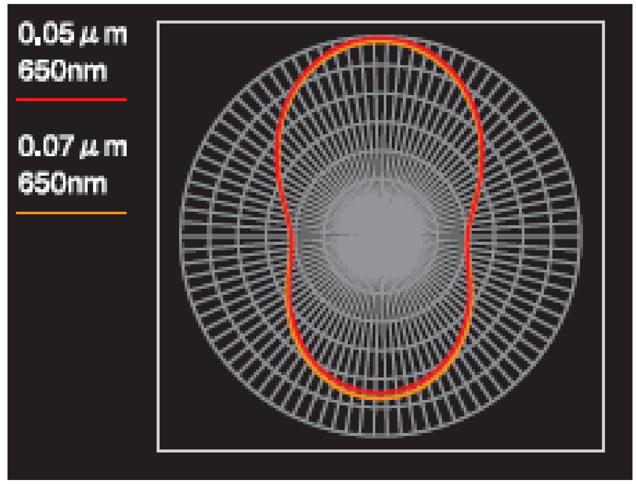

Figure 5: Light scattering patterns for 50 nm and 70 nm particles using a 650 nm laser.

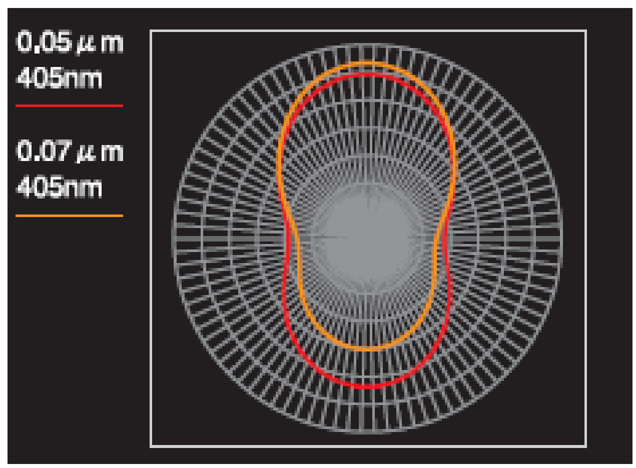

Figure 6: The light scattering patterns for the same samples using a 405 nm LED.

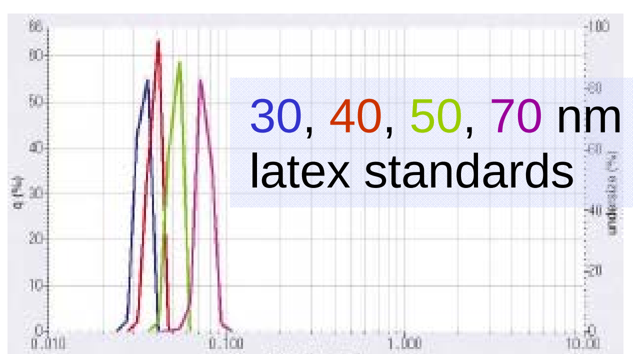

Figure 7: 30, 40, 50, and 70 nanometer materials independently measured on the LA-950 using the blue LED

Technical Note

LA-950 Technique

Summary

The HORIBA LA-950 particle size analyzer uses the laser diffraction method to measure size distributions. This technique uses first principles to calculate size using light scattered off the particle (edge diffraction) and through the particle (secondary scattering refraction). The LA-950 incorporates the full Mie scattering theory to cover the widest size range currently available. Wide measurement ranges, fast analyses, exceptional precision, and reliability have made laser diffraction the most popular modern sizing technique in both industry and academia.

References

1. ISO 13320, Particle size analysis -- Laser diffraction methods -- Part 1: General principles
2. "Understanding Calculation Level and Iterative Deconvolution." Horiba.com/us/particle, 2007

For further information on this document or our products, please contact:
HORIBA Instruments, Inc.
34 Bunsen
Irvine, CA 92618 USA
1-800-4-HORIBA
www.horiba.com/us/particle
labinfo@horiba.com