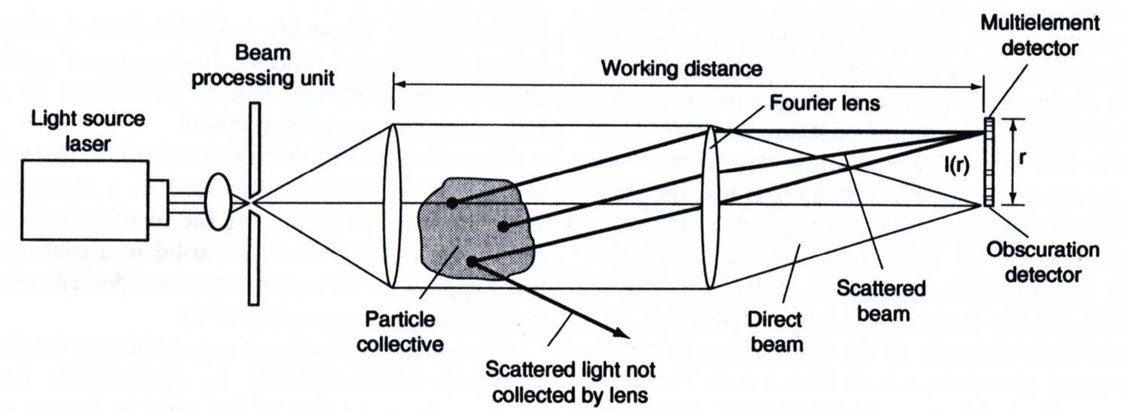
OPTIMIZING MEASUREMENT OF SUB-MICRON PARTICLES WITH LASER DIFFRACTION

Early laser diffraction particle size analyzers were of limited range and the designs from different manufacturers was similar. With the drive to widen the measurement range, different approaches have been taken, particularly for measurements in the sub-micron range.

Early Instrument Designs

As laser diffraction instruments were developed from their basic origins, different approaches and designs have become available. The basic laser diffraction instrument design uses a laser light source, a beam processing unit (usually a beam expander and collimator) to increase the cross section of the light beam, the measurement zone, a lens to focus the light on the detector, and a multi-element detector. The working distance from the particle to the collector lens defines the range of the instrument. Shorter working distances will measure a smaller particle size range than a longer working distance.

Various size ranges are accomplished by changing focal length and position of collector lens. An attempt is made to capture most of forward scattered light by locating the lens very close to the sample cell. The limitation is the relatively narrow size range that can be measured at one time and the danger of missing detection of particles beyond the active range.

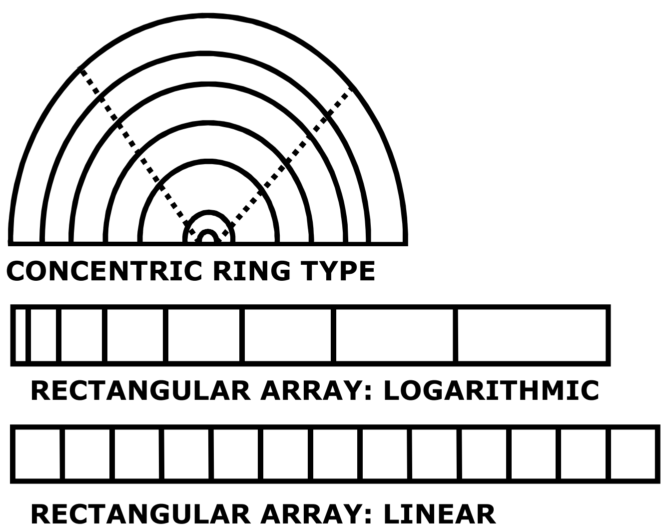


Detector System Design

When the detector system is designed, the behavior of light must be considered. As particle size decreases, the angle of scatter increases logarithmically and the intensity of the scattered light decreases. Taking this into account, the detector elements should be designed with a logarithmically increasing detector segment width and spacing.

If the detector has linearly spaced detectors, as is seen with off-the-shelf detector arrays, little additional information would be gained from the individual narrower detectors at the higher angles. Instruments that use this detector type often add signals from several detectors together to get a corresponding angular range, at the cost of adding the noise from these multiple segments. The result is a decreased signal to noise ratio compared to a properly designed detector.

Historically, as companies attempted to increase the dynamic measurement range for an instrument design, the options were either to increase the size of the array, which is expensive, or to add additional detectors. A number of companies added a second detector array, usually the same part as the forward scatter array. While this was an economical approach, the forward array with its narrow detector segments is less than ideal for measuring the higher-angle, low-intensity scattered light. An alternate approach is to use a second light source at an angle to the cell, so that the scattered light collected on the main detector is from the higher angle scatter. Either approach has the same limitations of an inappropriate detector design for the task.



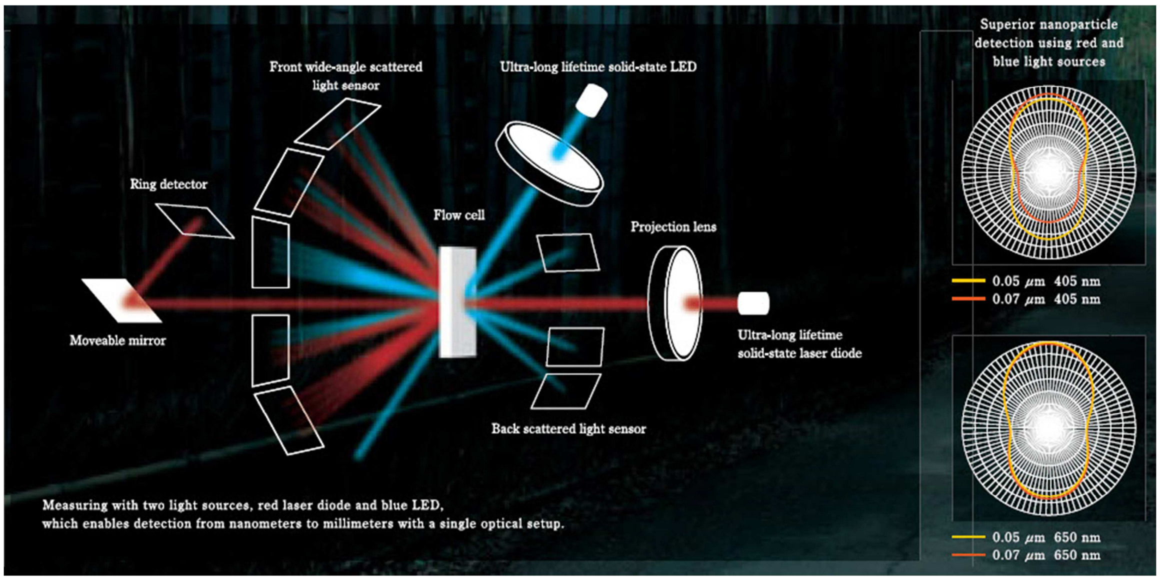
In HORIBA's LA-series analyzers, the detector is a fan shape with logarithmically increasing segment spacing and a much larger surface area for the low intensity light scattered at higher angles. This helps compensate for the lower intensity light without having to boost the electronic gain and possibly electronic noise.

<u>Improving Sub-Micron Measurement</u>
To extend the particle size measurement range into the sub-micron range, several companies have tried to marry different analytical approaches to angular light scattering, although only one company remains with this approach. The limitations are due to how to accurately blend two different techniques that may not have an equivalent response. Difficulties are seen particularly in the crossover area between the two techniques. Attempts are also made to extend the basic Fraunhofer approximation to these lower sizes.

The most complete and consistent approach that will work with the ability to measure large particles is to extend the full Mie Theory measurements to the smaller sizes with an appropriate optical system. At the smaller sizes, the wavelength of light becomes critical. The shorter the wavelength of the incident light, the greater the intensity of the scattered light will be. The detector system must be arranged to account for the logarithmic increase in angle and provide sufficient coverage of the angular range to accurately measure the scattering pattern from smaller particles.

HORIBA's approach uses two light sources, a 650 nanometer solid-state red laser diode and a 405 nanometer solid-state blue LED. The laser is used for both forward scattering and for the high angle detectors. The blue LED is only used for high-angle scattering. The combination of both wavelengths provides a full picture of the scattering pattern for sub-micron particles, together with sufficient intensity, due to the shorter wavelength, to obtain a high signal-to-noise ratio. The detector system is a combination of a logarithmic detector array and logarithmically spaced high-angle and back-scatter detectors.

The benefits from this approach are multiple. Mie Theory is used to cover the entire size range, the optical system is a logical progression from large particles to small and there is one sample handling system, cell and detector system for all sizes of particles. The measurement is a seamless transition across the entire range and the sample handling systems are consistent for all types of materials.



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