New Applications and Recent Developments of Particle Size Distribution Analyzer using the Laser Diffraction Technique

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The LA-960 particle size distribution instrument which uses laser diffraction/scattering was released in 2013. This measurement method can analyze many particles simultaneously in a short period of time, without contact, with high precision and repeatability. The LA-960 has accessories for several types of samples such as concentrated slurries, i.e. CMP, water soluble powders such as pharmaceuticals, agglomerated powders such as magnetic materials and small volume samples. We will discuss the features of the instrument and accessories in detail. We will also provide actual measurement examples to illustrate the instrument performance. Recently, the LA-350 particle size distribution instrument featuring laser diffraction/scattering was also released. We will discuss the features of the instrument which include a reasonable size measurement range and high performance in a very compact product.

Introduction

The particle size of many kinds of powders is an important factor in characterizing the product from an industrial process. The particle size distribution results from laser diffraction/scattering are used to control product quality for research and development in a wide number of fields such as ceramics, pigments, battery materials, catalysis, cosmetics, food and pharmaceuticals. When the powder market is examined, recent developments in nano-technology have evolved to make finer particles or to combine multiple materials together more common. This has led to the market requesting more accurate measurement of particle size distributions of very fine particles.

The Partica LA-960 (**Figure 1**) utilizes a red laser diode and blue LED which is able to collect scattering patterns as a function of angle. The Partica LA-960 is widely accepted to measure particle sizes less than 1 μm with high accuracy and good repeatability. Many detectors located around the cell detect the scattered light intensity pattern. The calculation of particle size distribution is determined by minimizing the difference between this scattering intensity pattern and the theoretical pattern calculated by Mie theory[1]. In the case of such a non-linear problem; there are many possible answers from one scattering intensity pattern. It is important to select the proper input parameters combined with knowledge of the particles being examined. The instrument has an ultrasonic probe to disperse aggregated particles to primary particles. The pump in the circulation system is able to select up to 15 different circulation speed settings, which allows it to circulate even Zirconia ceramic balls at 3 mm diameter, and a density of 6.0 kg/m^3. For emulsions that are likely to foam, the software is able to reduce the speed of the circulation pump. The circulation system is able to disperse homogeneously several kinds of particles with large size, high density or in a flocculated state. The circulation system contains an agitator to disperse many kinds of particles, even those with low density and small size which tend to float on the top of the liquid in the sample bath. Many accessories and options are available to disperse difficult samples. The features of the accessories will be discussed and the measurement results for some important applications presented.

Recently, the LA-350 particle size distribution instrument also featuring laser diffraction/scattering was released (**Figure 2**). The LA-350 features the LA-960 performance in the form of a

Figure 1 LA-960

Figure 2 LA-350

reasonable measurement size range and high performance in a very compact product.

Principle

Laser diffraction/scattering [2] is a type of measurement method for particle size utilizing the scattered light intensity pattern as a function of the scattering angles. The scattering light intensity pattern is changed by the relative difference between the particle size and by the effect of the wavelength of irradiating light. The diffraction is dominant for particles with a size greater than 10 µm. For smaller particles the scattering in the reverse direction predominates. When the particle size is smaller than 10 µm, the scattered light intensity pattern is dominated by Mie scattering which is influenced by the relative refractive index difference between particle and dispersant. The scattering intensity pattern is seen in 180° of scattering. This light must be detected by a wide range of detectors including side scattering angles and back scattering angles. When the particle size is below a 1/10 of wavelength of light source, the scattering light intensity pattern does not change significantly. This phenomenon is known as Rayleigh scattering. There is no difference caused by the decreasing particle size; therefore this theory cannot be used for the scattered light intensity pattern. The LA-960 uses Mie-scattering theory for the calculation of all particle size distributions. Mie theory covers the diffraction and scattering range of particle size from nanometer to millimeter, and the method is able to analyze this particle size distribution range with good accuracy.

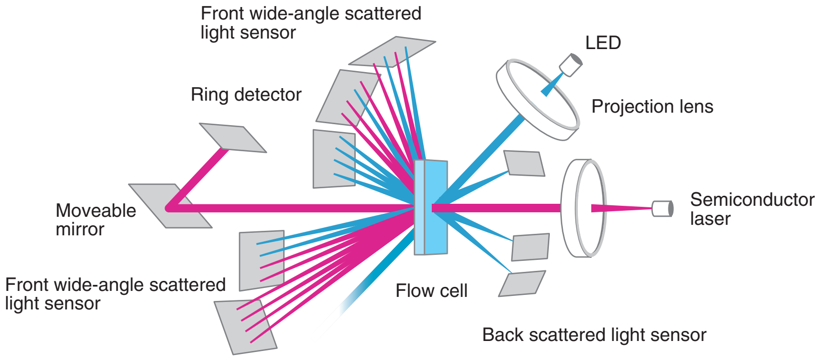


Figure 3 LA-960 optical system

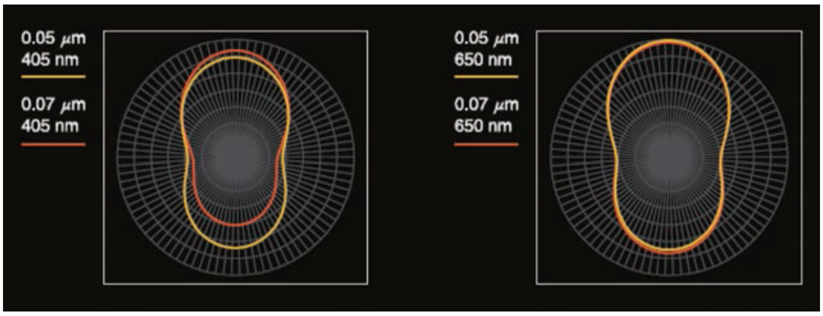


Figure 4 Changes in scattering patterns from microscopic particle diameters

Partica LA-960

The key features of LA-960.

Optics

Figure 3 shows the optics diagram for the LA-960. The light from the laser diode at 650 nm is focused on the particles circulating in the flow cell. The diffracted light is scattered to the ring detector for forward scatter and at the side or back scattering detectors covering all 180°. The light from an LED at 405 nm is also focused on the particles circulating in the flow cell, and scattered light from the side or back scattering detectors again cover a large series of angles. This light source is for the measurement of smaller particles below 1 µm. The two light sources of the optics system make it possible to measure particle size with wide range from nm to mm. **Figure 4** shows the scattered light intensity patterns for a 50 nm particle, and the other is the pattern for a 70 nm particle. When we compare the scattering pattern between the 650 nm and 405 nm sources, the scattering pattern from the 405 nm source is able to show differences in the scattering pattern from both particles. The 405 nm source shows a difference, making it possible to measure the smaller particles. This is one of the features on the LA-960 that permits measurement of particle sizes below 100 nm accurately.

Circulation system

Figure 5 shows a diagram of circulation system. The circulation system consists of a sample bath, a flow cell, and a circulation pump. The pump is designated to circulate samples from the bath to the cell without segregation, adhesion to the tubing or build-up in the system. A fluid sensor detects the liquid level in the bath, the ultrasonic probe disperses agglomerated particles to primary particles, and the drain valve permits removable of the sample following measurement. The liquid volume is able to be

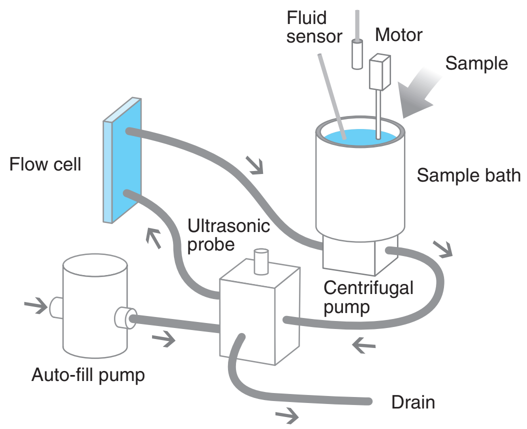


Figure 5 LA-960 circulation system

controlled from a minimum of 180 ml to a maximum 280 ml. The LA-960, for one sample measurement including feeding the liquid, dispersing the particles, measurement, drain, and rinse of circulation system, can be completed in 60 seconds.

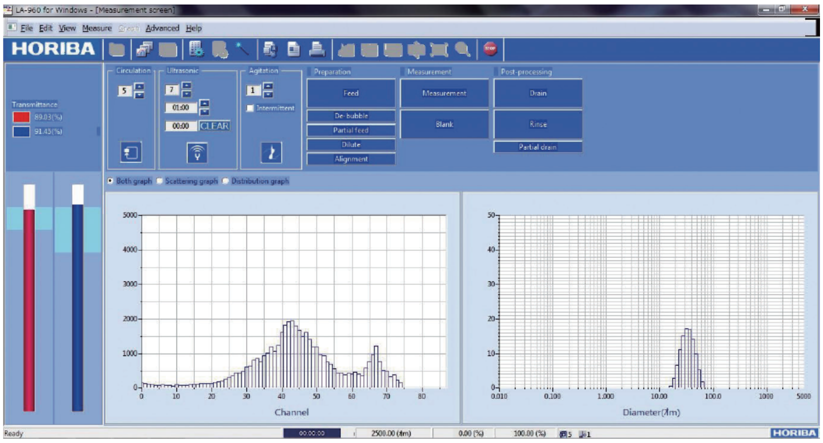


Figure 6　Measurement window

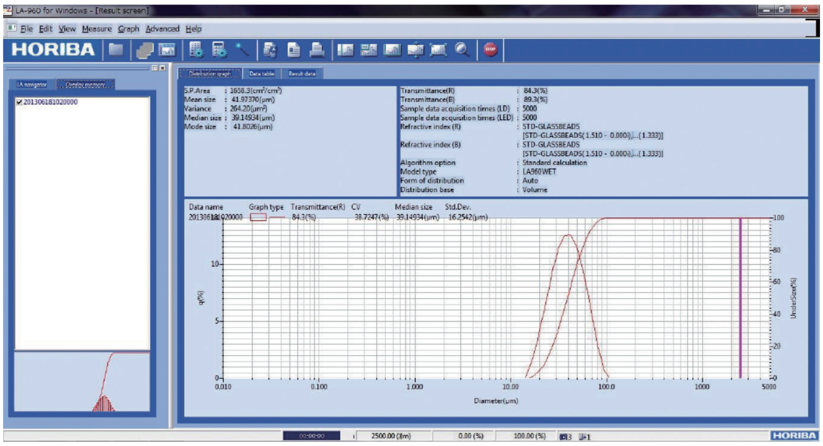


Figure 7　Measurement result window

Software

Figure 6 shows the measurement display. There are two colored bars with the red bar showing the transmittance (%T) of the red laser diode, another blue bar showing the transmittance (%T) of the blue LED. When a sample is placed in the bath, the transmittance %T is examined in order to adjust sample concentration. There are two graphs at the left side of the display showing scattered light intensities for each detector channel, and the right graph showing the real time display of the particle size distribution. The real time monitor is valuable if the particles require dispersion. It can detect if some aggregated particles are remaining in the sample or if some bubbles have been introduced. The LA-960 dispersion conditions can be adjusted to remove bubbles or to disperse hard agglomerates by confirmation with the real time monitor. **Figure 7** shows typical measurement results. The size distribution can be displayed in several formats, for example, volume based distribution, number based display, ISO format, cumulative displays and custom formats. The upper portion of the display shows the measurement conditions and measurement results for the active data. The LA-960 software has several kinds of analysis tools in the advanced analysis module. Here, there are several menus that display the scattered light intensity pattern or intensity value for each detector, and also de-convolution tools for the distribution peak. Using these tools properly are key features to analyze the size distribution properly.

Accuracy

It is difficult to detect scattered light from nanoparticles, because they scatter the light from the particles at all 180° degrees and provide a weak signal. With the LA-960 it is possible to measure below 100 nm. The LA-960 guarantees the nominal diameter ±0.6% accuracy for 8 sizes of Polystyrene latex NIST traceable standards. The 8 standards are used as calibration particles ranging from 20 nm to 5000 μm, prior to shipment from our factory. The LA-960 is further tested for performance such as repeatability and accuracy based upon ISO13320 by testing a wide size distribution of traceable glass beads.

Accessories

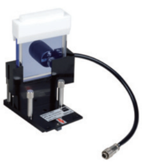
Figure 8 shows several kinds of accessories to facilitate measurement possibilities. They are the Dry Powder Measurement unit, the Miniflow, the Fraction Cell unit, the Paste Cell unit and the Auto-sampler.

Dry measurement unit

This unit is used to evaluate the particle size distribution of dried powder, for example, pigments, foods, medicines or sands. The unit is able to measure the size of powders without dispersion in a liquid system. The powders on the feeder tray vibrate to the

Dry unit

Mini flow



Fraction cell

Paste cell

Auto sampler

Figure 8　Accessories

entrance of the cell, and sample is dispersed by pressurized air in the cell, and the sample and air are sucked up into a vacuum cleaner. The dry powder flow system prevents the dispersed particles from agglomerating again. This unit has convenient auto-measurement functions, which speed up the analysis. One is auto-trigger that automatically starts the measurement when the transmittance signal indicates sample flow is correct. Another is the control of the amount of sample by selection of the feeder speed. These conditions are selected based upon the properties of the powder such as material type, viscosity of the powder flow or amount of sample available for analysis.

Miniflow

This unit is designed to permit the use of organic solvents with a minimum volume of 35 ml. The unit is able to disperse agglomerated particles with ultra-sonication and to circulate the sample into the sample cell for measurement. Some applications are dispersed products in an organic solvent, oil or special expensive solvent. Examples include pigments, paint materials, inks, medicines and battery materials.

Fraction cell unit

This unit is available to use organic solvents with a solvent volume of a minimum of 5 ml. It is also possible to recapture the sample after measurement. Most typical applications include dispersed products in the highly-volatile solvents, expensive rare earth metals or pharmaceuticals.

Paste cell unit

This unit is for measuring concentrated samples without dilution. A typical example is a readily agglomerated sample such as magnetic materials. These samples are dispersed in a high viscosity solvent such as silicon oil, and the sample is sandwiched between the two cell windows to prevent agglomeration. These accessories are typically used for samples at high concentrations and high viscosity, or high concentration and low viscosity. The later one requires use of a syringe to fill the sample within the space between the two cell windows. Most applications are for dispersed products at high concentration, for example, inks, pigments, surfactant particles in polymers and slurries.

Auto-sampler

This unit automates the analysis by the use of a rotating table including 24 sample cups. The unit feeds samples one by one into the sample bath, and then adds rinse water into the cup holder. Applications include products which are a part of a quality control program, such as ceramics, electronic parts, coatings and catalysis.

Measurement results

The features of LA-960 are covered below.

Measurement results of mono-dispersed samples

Figure 9 shows the measurement results for 30 nm, 40 nm, 50 nm and 70 nm polystyrene latex standards. HORIBA's optical technology and signal processing technology makes it possible to detect low intensity signals from small particles below 100 nm with high sensitivity and good repeatability.

Figure 10 shows the measurement result for a 4 mm glass beads standard. The LA-960 optics features a long focal length lens system. Therefore, it is possible to detect low angle signals from large particles in the mm range with high sensitivity and good repeatability. This result comes from the gravity feed dry cell.

Figure 11 shows the measurement result of a broad sample composed of a mixture of two alumina samples with different size ranges. The one has a median of 1.3 µm, and the other is 6.2 µm. The result clearly shows the distribution for both peaks.

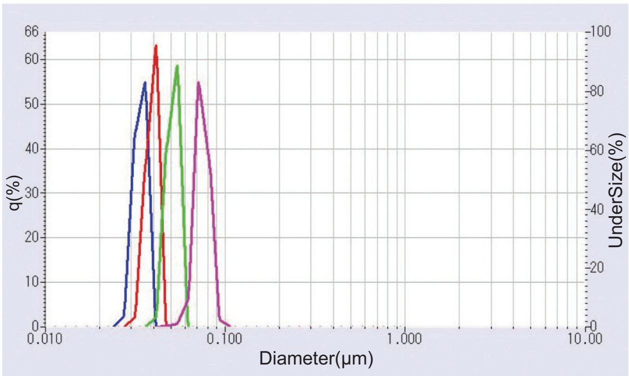


Figure 9 Polystyrene latexes (NIST Standard)

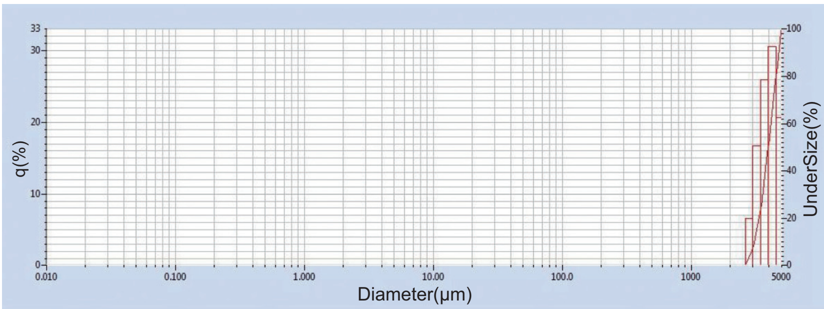


Figure 10 Glass beads(4mm)

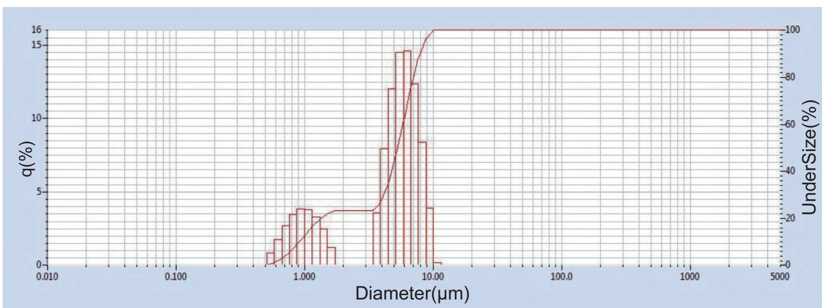


Figure 11 Bimodal Alumina (WA#2000: D50=6.2µm, WA#8000:D50=1.2µm)

Figure 12 shows a measurement result for a bacteria sample. The bacteria fluoresced at high concentrations was measured at the high concentration in the low viscosity cell with a sample volume of only 0.2 ml. The median size was 2.2 µm. It is possible to measure biological samples without dilution; to prevent modifying the sample's properties.

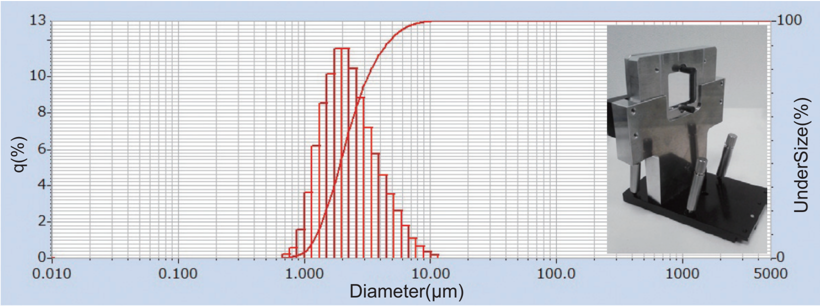


Figure 12 High concentration & Low viscosity cell

Figure 13 shows the measurement result for a concentrated dye-ink used for a printer application. The samples were measured at high concentration in a high viscosity cell; with a sample volume was only 0.1 ml. It was also possible to measure magenta ink and yellow ink without dilution, and cyan ink and black ink with a dilution of less than 10 fold.

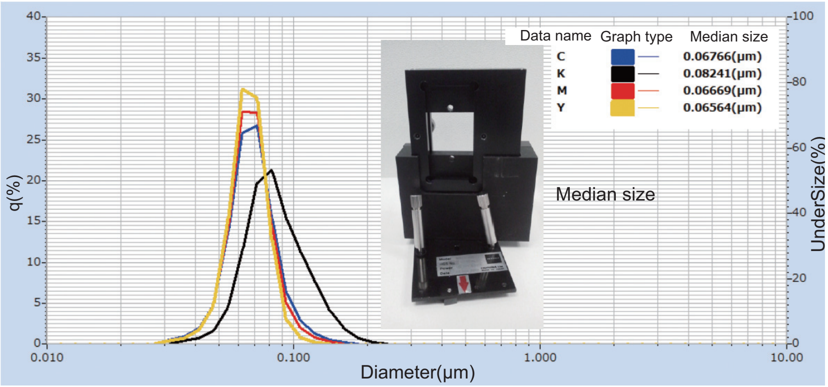


Figure 13 High concentration & High viscosity cell

Partica mini LA-350

The key features of LA-350.

The LA-350 uses the same fundamental design plus, adds a reasonable size measurement range and high performance within a very compact product. The instrument includes a powerful pumping system and a probe-type ultra-sonication system with similar performance to the LA-960. The measurement range is expanded to 0.1-1000 µm relative to the LA-300. The maximum range has been expanded from 600 to 1000 µm without the need for a new optical design. The LA-300's detectors are increased from 42 channels in the LA-350 to 70 channels. All LA series analyzers use the same software, so that all users can easily switch from one instrument to another.

Conclusion

The features of Partica LA-960, the accessories and latest applications have been described. In addition, the features of the LA-350 have been introduced. These two instruments should prove to be valuable tools for researchers who are examining ever smaller particles.

References

[1] M.Umezawa, H.Sugasawa *Readout*, **41**, 92 (2013).
[2] K. S. Shifrin, et al., *Atmos Oceanic Phys.* **8**, 739 (1972).

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