Product Introduction

The LA-960 Laser Diffraction/Scattering Particle Size Distribution Analyzer

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The LA-960 particle size distribution measurement instrument with laser diffraction/scattering is a next-generation model that was developed as the highest-end model of its type. The LA-960 is based on the LA-950V2 hardware design, which has a high level of design maturity and has been popular in the past, and has significantly improved analysis capabilities compared to that model. The following 5 points are features of the LA-960.

1. Improved minimum particle size measurement accuracy and reproducibility
2. Increased maximum measurable particle size
3. Introduction of the latest calculation algorithms
4. New software GUI
5. Data compatibility with the old model

We would like to discuss the features of the instrument in detail. We will also provide actual measurement examples to show instrument performance.

Introduction

The size of powder particles used in various industrial processes is an important element that defines product functionality. HORIBA's laser diffraction/scattering particle size distribution analyzer is used for the objective of research and development and quality control in a wide variety of fields, such as ceramics, pigments, battery materials, catalysts, cosmetics, food products, and pharmaceuticals.

If we look at particle market trends, we can see that recent developments in nanotechnology have engendered remarkable progress in technologies for reducing the size of particles and composing particles, and there is an increasing need to be able to measure fine particle diameter distributions with higher accuracy. The LA-950V2 had a very high level of maturity in terms of hardware performance, and was highly valued. In developing this device, HORIBA had used remarkable recent developments in computer technology to significantly improve data analysis performance. The LA-950V2 also had improved capability for analyzing optical signals it detected, and was described on Page 93 of Technical Report No. 41 (September 2013). The Partica LA-960 (Figure 1) is a next-generation laser diffraction/scattering particle size distribution analyzer with the latest data analysis technology and significantly improved analysis capabilities.

Figure 1 LA-960

Measurement Principles

The laser diffraction/scattering method uses differences in scattered light patterns that change based on particle diameter to find the particle diameter. When incident light at a certain wavelength is aimed at a single spherical particle, the scattered light intensity distribution changes based on the relative particle diameter with respect to the incident wavelength.

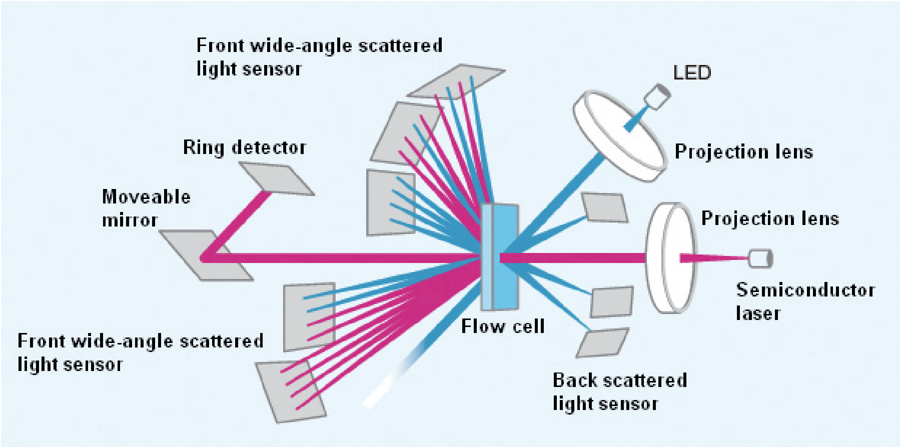

Figure 2 LA-960 optical system

When a particle has a diameter of about 10 µm or more, edge diffraction is dominant. Scattered light is concentrated in the forward direction, which is the direction that incident light passes through, and the diffracted light intensity distribution is a mathematical function of only the particle size. When a particle has a diameter of about 10 µm or less, the relative index of particle refraction causes the scattered light intensity distribution to be a Mie scattering pattern, which changes with a high sensitivity. Scattered light is detected not only in the forward direction, but in a wide angle range, from the side to the rear. When a particle is even smaller, and has about 1/10 or less of the wavelength, the change in scattered light intensity distribution is small, even if the particle diameter changes. This scattering pattern is known as Rayleigh scattering. It is difficult to identify the particle diameter based on the scattered light intensity distribution, and this is the lower limit of the measurement principle.

Things like diffraction and Rayleigh scattering are simplified as scattering phenomena. Using the Mie scattering theory for scattering phenomena makes it possible to analyze a wide range of particle diameters with a higher accuracy, and the LA-960 uses these Mie scattering theory calculations.

Device Overview

We would like to introduce new features of the LA-960.

Improved accuracy for measuring minimum particle diameter

In addition to the wide measurement range of the dual wavelength optical system that was established by previous models, HORIBA developed new signal processing algorithms for measuring weak light, and the LA-960 has a high measurement accuracy of ±0.6% for standard polystyrene latex particles with particle diameter, 20 nm. This represents industry-best performance.

Expanded upper limit for measurable particle diameter

Figure 2 shows the optical system configuration. The LA-960 effectively uses the long optical path length between the flow cell and ring detector to achieve a high spatial resolution. This makes it possible to reliably detect low-angle scattered light signals that are sensitive to large particles with high accuracy. The LA-960 has achieved a wide measurement range, up to 5000 µm. Also, the optical system detector is configured in the height direction, which ensures that the device takes up almost the same floor space as the previous model, making it attractive for users with limited lab space.

Most advanced calculation algorithms

The LA-960 uses the latest theories for data analysis and newly developed calculation algorithms, and provides stable, high-quality analysis performance for all particle diameters. Also, the new calculation algorithms eliminate the need to enter the number of iterations, which was required on previous models. When the particle diameter distribution calculations are run, the optimal calculation conditions are automatically set.

New software GUI

The LA-960 software GUI has been improved in many ways. The software design has been updated, and the interface is now more intuitive. Buttons and icons that are used very frequently have been reconfigured to provide better practical use. Buttons that are used very frequently are displayed larger with a larger font size, and are easier to see.

Figure 3 shows the measurement screen, and a simple calculation of scattered light intensity and particle diameter distribution are displayed in real time. Whether or not there is particle agglomeration or air bubbles can be checked on the main screen, so that users can take measurements in the optimal dispersion state, By checking for agglomeration and air bubbles, users can optimize the dispersion state by running ultrasonic dispersion and air removal sequences.

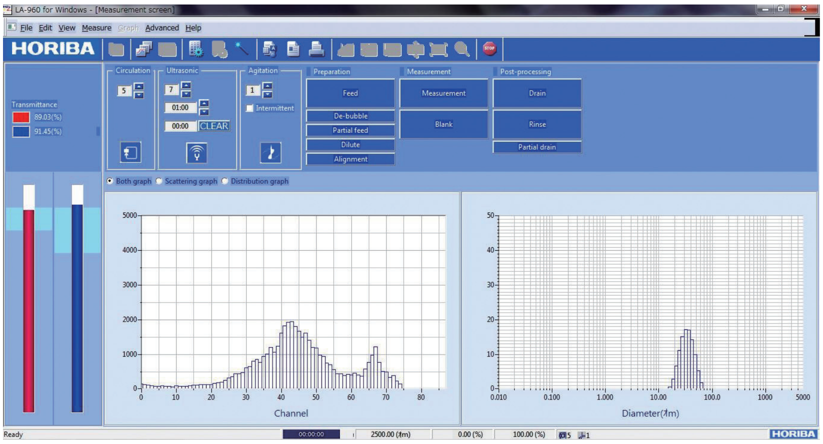

Figure 3 Measurement window

Figure 4 shows the measurement result screen. For particle diameter distribution, users can choose from a variety of displays, such as graph format or cumulative distribution. Information on measurement conditions and measurement results is displayed at the top. As expanded functions, tools for analyzing the measurement results using a variety of methods have been provided, such as scattered light analysis, detector output confirmation, and distribution peak separation tools. Users can do various types of analysis by using these functions effectively.

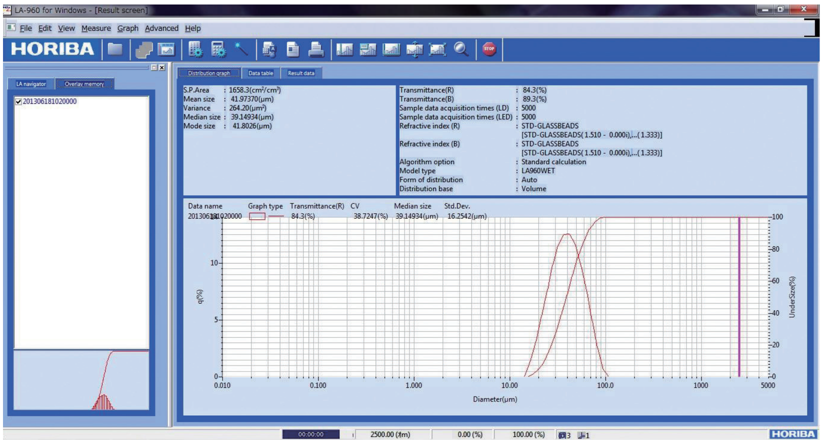

Figure 4 Measurement result window

Data compatibility with old models

The LA-960 software supports full data compatibility with the LA-950V2 model (one generation before the LA-960) as a standard specification. Software with a data correlation function for the LA-920 model (one generation before the LA-950V2) can be provided for a fee.

Improved Particle Diameter Distribution Analysis Performance

The laser diffraction/scattering particle size distribution analyzer detects scattered light signals, analyzes them based on the Mie scattering theory, and calculates the particle diameter distribution. The LA-960 has high-speed data processing and uses the latest calculation theories, taking advantage of recent advances in computer performance. The next section will explain some of those details.

Next-generation analysis algorithms

With static light scattering particle size distribution measurement devices, when particle size distribution is calculated, inverse problems need to be solved for lines. The previous LA-950 model used an old calculation method developed in the 1960s called the Twomey iteration method. However, mathematically, solving inverse problems of matrix is similar to Internet search problems and analyzing biological signals in many ways, and various solutions have been developed recently. The LA-960 applies these solutions to distribution calculation problems, and has achieved better peak location accuracy and fewer false solutions than was possible with the Twomey iteration method. Specifically, the LA-960 calculation algorithms use multiple solution methods, such as the symmetric LQ method, modified residual norm steepest descent method (MRNSD method), and the hybrid bidiagonalization regularization method (HyBR method).

High-accuracy Mie scattering theory function

To accurately calculate particle diameter distribution based on detected scattered light signals, a high-accuracy Mie scattering theory function that corresponds to the measurement sample’s relative index of refraction is required. This time, to increase the accuracy of the theory function calculations, the data length was increased. More specifically, we changed the base number used in the function from double (64-bit primitive floating point) to BigDecimal (arbitrary precision signed decimal). This made it possible to minimize the cancellation of significant digits and loss of trailing digits that occur in infinite series calculations run during particle diameter distribution calculations and ensure sufficient calculation accuracy (same accuracy as display area). However, there was a concern that increasing the amount of data handled and object generation time would cause a delay in Mie scattering theory function calculation time. We solved this problem by using concurrent calculation processing and utilizing the cache to improve the efficiency of calculations and achieve a higher calculation speed. It is possible to create a high-accuracy Mie scattering theory function in the same time as the current model.

The LA-960 uses ray tracing technology based on 3D modeling in a test environment that can uniformly handle things like cells, slits, detectors, and scattered light sources to evaluate the ideal Mie scattering theory function. The LA-960 Mie scattering theory functions precisely take things like the effects of detector and light source mechanical errors and the effects of reflected light inside and outside the cell into account, and contribute to accurate measurements (Figure 5).

Examples of Measurement Results

We would like to provide a few measurement examples to demonstrate the measurement performance of the LA-960.

Example of measuring a monodisperse sample

Figure 6 shows the results of 3 consecutive measurements of 20-nm standard polystyrene latex particles near the lower limit of measurable particle diameter. All 3 measurement results have a measurement accuracy within ±0.6%. Weak light signal processing performance has been significantly improved, making it possible to measure 20-nm polystyrene latex particles with high accuracy and high sensitivity.

Figure 7 shows the results of measuring a glass bead with a diameter of 4 mm near the upper measurable limit. The LA-960 has improved weak light signal analysis performance and an expanded upper measuring limit that provide a wider range of higher-accuracy measurements than previous models.

Example of measuring a polydispersed sample

Figure 8 shows an example of measuring multicomponent

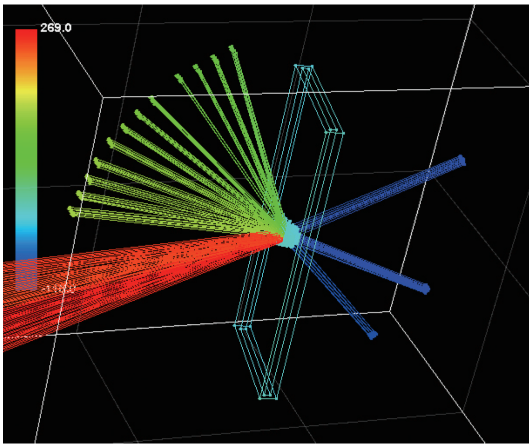

Figure 5 3-D ray tracing-applied Mie scattering modeling

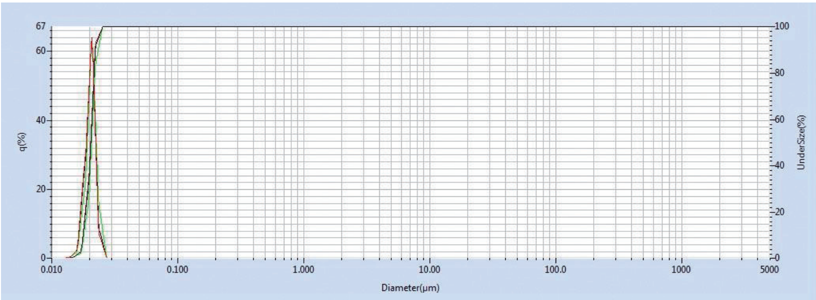

Figure 6 20 nm polystyrene latex (NIST Standard)

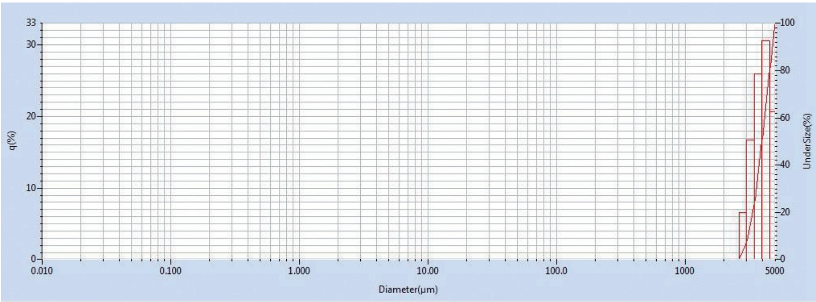

Figure 7 Glass beads (4 mm)

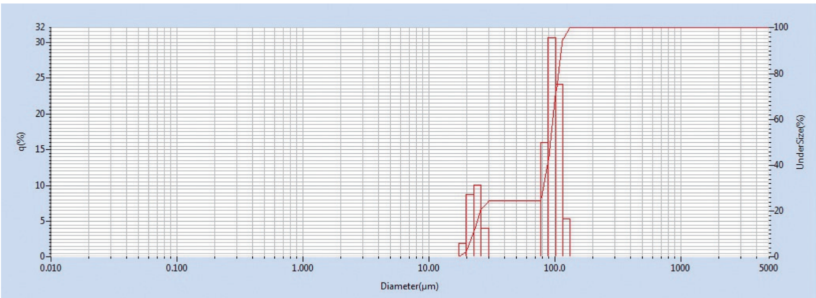

Figure 8 Bimodal glass beads (GBM-20 : D50=21~23 μm, GBL-100 : D50=99~101 μm), mixing ratio 3 : 7 (GBM-20 : GBM-100)

mixture samples. The LA-960 can measure the particle diameter peak location and mixing ratio of mixed particles with high accuracy. Figure 8 shows the results of measuring particle diameter distribution of a mixed sample of 2 types of glass beads. In terms of peak location and peak height, accurate distribution peaks can be obtained for glass beads with respective diameters of 22 µm and 100 µm, and an accurate peak height can be obtained for a mixing ratio of 3:7.

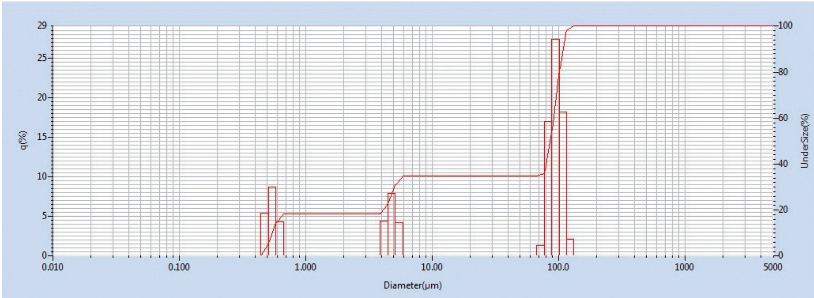

Figure 9 Trimodal PSL (0.08 µm, 6 µm, 100 µm)

Figure 9 shows the measurement results for 3 different types of particle diameter distribution. This is one example showing the results of peak resolution height.

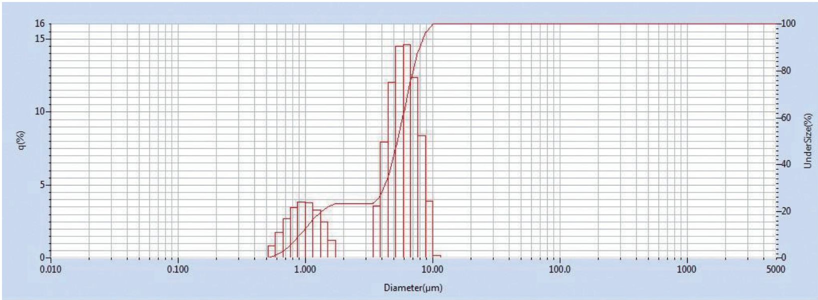

Figure 10 Bimodal Alumina(WA#2000 : D50=6.2 µm, WA#8000 : D50=1.2 µm)

Figure 10 is one example of measurement results for a mixed sample with a broad distribution. Accurate respective particle diameter distributions have been obtained for mixed alumina particle diameter locations 1.2 µm and 6.2 µm.

The LA-960 provides high-quality particle diameter distribution measurement performance for a variety of particles in various distribution formats.

Conclusion

This paper introduced the features of the LA-960 and the continued state of the art development of calculation and analysis technology. In devices with optical systems, circulation systems, and calculation systems, calculation systems that calculate particle diameter distribution based on scattered light signals are a very important element, particularly in determining device performance. The LA-960 combines software with the latest analysis technology and reliable, high-performance hardware based on our years of experience with previous models. We hope that this device will meet customers' requirements for measuring particles with increasingly smaller particle diameters, aiding in quality control applications and other scientific and industrial applications.

Acknowledgements

Many people in Japan and overseas cooperated with us on the development and evaluation of the LA-960, and gave us a lot of very important advice. The HORIBA Group would like to take this opportunity to extend our sincere thanks to all those who helped. We hope that the LA-960 will be useful in a wide range of fields, from users' daily quality control to research and development.

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