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Particle Size Distribution Analyzer: Model LA-920

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一/般/論/文
Selected Topics

Particle Size Distribution Analyzer: Model LA-920

Terry STAUFFER* and Tatsuo IGUSHI

<Abstract>
The new Horiba LA-920 particle size distribution analyzer was designed to meet the present needs in particle measurement with significant advances in the hardware technology and in the software - in both the algorithms employed and in the automation and operational conveniences that were added Measurement performance, in terms of resolution, dynamic range (0 02 to 2000 microns), repeatability and reproducibility, have all been improved with a new 87-element detector system, auto-alignment and an advanced iterative algorithm that optimizes the resolution A new circulation system features a centrifugal pump, solvent-compatible surfaces, and an ultrasonic probe in the flow stream for maximum particle dispersing capability The operation of the analyzer is much simplified with automatic features that minimize operator involvement, and data retrieval and manipulation capability is enhanced with a new data base management system

1. Introduction

The new Horiba LA-920 particle size distribution analyzer was designed to meet the present needs in particle measurement with significant advances in the hardware technology and in software, in both the algorithms employed and in automation and conveniences

* HORIBA Instruments Inc

粒度分布測定装置 LA-920

1　はじめに

粒度分布測定におけるユーザーニーズの傾向は，分解能，ダイナミック・レンジ，再現性等を含む測定性能，最高の粒子分散，自動化，操作性などに顕著である．堀場の粒度分布測定装置LA-920はこれらのニーズに対応し，新たに設計したハードウエアおよびソフトウエアによって，粒度測定における光散乱/回析法の有用性を実証した．

2　測定性能

2 1 分解能

LA-920によるNISTラテックス標準試料0 2，0 3および0 5ミクロンの混合サンプル(図１)，ラテックス標準試料0 7および1 0ミクロンの混合サンプル(図２)の測定結果を示す．この分解能は，第一に広角検出器の数を2倍にし，第二に2種の異なった波長の散乱光により検出情報を増加し，第三に検出アレーを含む検出

1 1 Trends in User Needs in Particle Size Analysis

General user needs in particle size analysis have moved in the direction of improved measurement performance, optimal particle dispersing ability, automation, and operational conveniences Measurement performance includes such criteria as resolution, dynamic range, instrument repeatability and instrument-to-instrument agreement

1 2 Trends in Light Scattering Technology

Angular light scattering technology* has made advances in all these areas The need for measuring finer and finer sizes (increased dynamic range) was met by including Mie corrections in the deconvolution algorithms, which themselves have advanced from the early matrix inversion approach to iterative routines that maximize resolution Continuous advances in computers and software have allowed more and more automation of the measuring process as well as data and display manipulation conveniences for the operators

* Angular light scattering is based on the principle that particles scatter light at angles inversely proportional to their size, the smaller the particle the larger the angle of scatter As with all particle size measuring techniques, the method assumes that the particles are spherical. Factors affecting the measurement include shape, refractive index and sampling and dispersing techniques [1 2]

2 Measurement Performance Needs

2 1 Resolution

Resolution in particle size distribution analysis refers to the ability of the analyzer to resolve multiple peaks (modes) in the distribution and the extent to which separation occurs between adjacent peaks For example, two instruments may resolve a mix of two different narrow distributions, but one may go to baseline between peaks and the other only down to say 50% of the height of the peaks The instrument that goes to baseline between the two peaks would be considered to have better resolution, even though both identified the two modes correctly

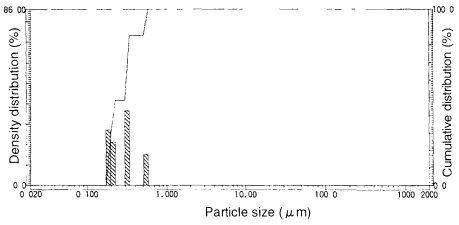

Fig 1 Measured result of mixed polystylen latex standard (0 2, 0 3, 0 5 μm)

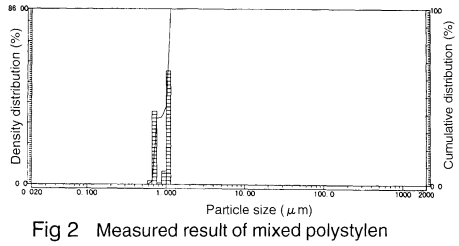

Fig 2 Measured result of mixed polystylen latex standard (0 7, 1 0m)

A high degree of resolution is desired to be able to detect minor but important differences in size distribution Often small amounts of a slightly coarser mode than the main mode indicates unwanted agglomeration, or stray particles that can damage surface finishes, by abrasives or in coating applications A small amount of an additional fine mode may indicate the small amount of active ingredient added to a pharmaceutical preparation, and therefore it must be measurable to maintain correct specifications

Figures 1 and 2 show some results of measurements on the new Horiba LA-920 particle size analyzer In Figure 1, a mixture of three NIST-traceable latex standards, at 0 2, 0 3 and 0 5 microns, demonstrates excellent resolution to

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器の素子総数を87個に増加して，達成された　さらにアルゴリズムの改良によって最適の分解能を得る反復計算の回数を自動的に決定するようにしている．

2 2 測定範囲

LA-920の測定範囲は，0 02～2,000ミクロン，ダイナミック・レンジは100,000である．後方散乱(図３)は，633nmのレーザ光に加えて，波長405nmの入射光により検出情報量を増加して0 02ミクロンまでの微粒子検出を可能にし，中心部に検出器を追加した．75素子のアレー検出器(図４)により，1,000～2,000ミクロンの範囲の粒子の検出を可能にしている

2 3 繰り返し精度

LA-920は，自動光軸調節装置により常に光軸がアレー検出器の中心に当るように設計されており，手動作業では達成できない高い繰り返し精度の保持を達成した

baseline between the peaks, which are separated by only 0 1 and 0 2 microns respectively Figure 2 shows a mix of two latex standards at 0 7 and 1 0 microns with the same degree of resolution Note also that each standard is reported at exactly its specified size in both graphs

This superior resolution is accomplished with a combination of features that work together to achieve the end result First, the number of wide angle detectors which detect the fine size range have been increased from six to twelve, providing more measured points in the range Second, each of these detectors measures the scattered light from two different wavelengths, which doubles the amount of information used in the calculation Third, the total number of detectors has now been increased to 87, which improves the resolution capability by a factor of more than three And fourth, an advanced algorithm that automatically determines the number of iterations to use to achieve the optimum resolution has been incorporated into the size calculations

2 2 Range

A wider range capability gives the user more flexibility in that one instrument can handle more applications, and that minimizes the amount of equipment the user needs to have for particle size analysis

The range of the LA-920 is from 0 02 to 2000 microns, giving it a dynamic range of 100,000; this is the largest dynamic range available with angular light scattering instruments It is made possible in the LA-920 by a combination of the 12 wide angle detectors, the 405-nm wavelength incident light source and the new 75-element forward detector array

The wide angle detector system senses scattered light up to 150 degrees from the forward direction, which contributes more information to calculation of the finest sizes (smaller particles scatter light at higher angles) The 405-nm wavelength, in addition to providing more information to enhance resolution, allows the LA-920 to sense the light from particles that are smaller than can be sensed by the 633-nm laser light, by a factor of 405/633; in other words, it can sense particles that are 405/633, or 0 64 times the diameter of the smallest particles sensed by the laser light This, combined with the wide angle detectors, is what gives the LA-920 the smallest low-end range in angular light scattering instrumentation Figure 3 is a diagram of the wide angle detector layout in the LA-920

The new 75-element silicon diode forward detector array, in addition to enhancing the resolution in the medium and upper size ranges, provides additional

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2 4 再現性

工場出荷時に，LA-920は装置全数をNISTの標準試料で校正し，装置間の差(RSD)を2パーセント以内としている　4台のLA-920についてNIST標準試料3種を測定した結果を示す(図５).

2 5 精度のトレーサビリティ

米国の製薬産業では測定機の検定内容がFDA(米国食品医薬品局)要求に適合しないと，新しいプロセスは稼動できない．出荷時にNIST標準試料で校正しているLA-920は容易にこの要求を充たす　1,000ミクロン標準試料による結果を示す(図６).

3　サンプル粒子の分散

サンプル中で一部の粒子が互いに凝集していたり，凝固しているような場合，目的により，測定に先立ってそれらを最適状態に分散させる必要がある．

detectors at very low angles to sense particles that range from 1000 to 2000 microns, effectively doubling the range of the new instrument See Figure 4 for a schematic of the forward detector array

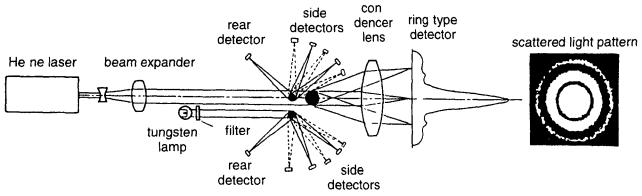

Fig 3 Wide angle detection system

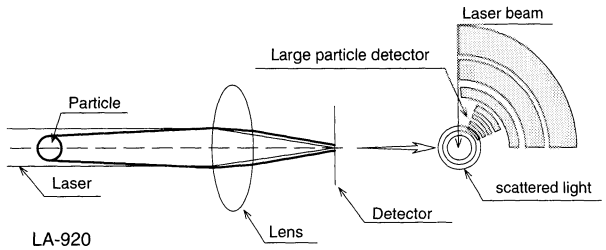

Fig 4 Forward detector array

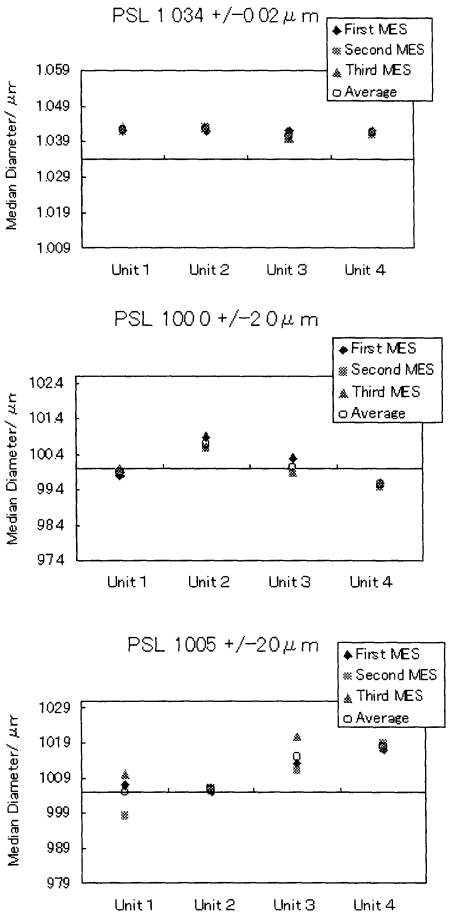

Fig 5 Reproducibility on four LA-920

2 3 Repeatability

The greater the instrument repeatability, the fewer the number of samples that need to be measured to achieve the same level of tolerance in statistical quality control applications[3] The LA-920 has a new auto-alignment system that automatically assures that the light beams stay targeted dead center on the forward detector array This allows the instrument to maintain a level of precision for each measurement, and therefore a level of repeatability, that could never be achieved manually

2 4 Reproducibility

Most companies have needs for more than one particle size analyzer, and most are involved in supplier-customer relationships for powdered materials This creates a very strong need for maximum reproducibility among instruments Customers need to verify specifications of incoming materials from their suppliers, and plants located around the world must get the same results from their instruments to insure company-wide product uniformity

The complete detector system, both the wide angle and forward array, of every LA-920, is factory-calibrated using NIST-traceable particle standards This standardized calibration assures a level of instrument-to-instrument reproducibility which results in less than a 2% Relative Standard Deviation (RSD) among instruments Figure 5 illustrates typical reproducibility on four production LA-920 analyzers Results are given for each analyzer on three different NIST standards (1,100 and 1000 microns) in a series of three measurements each

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3 1 分散エネルギーの設定

LA-920の循環システムに組み込まれている超音波プローブは，パソコンで必要な強さ，必要な時間等の条件を設定することにより，凝集･結合した粒子を任意のエネルギーで分散させることが出来る．

3 2 耐薬品性

循環システムの接液部は，ステンレス鋼，ガラス，テフロンまたはチタンである．これらの材質により，ほとんど全ての溶媒で問題なく使用できる．

3 3 測定セルにおけるサンプルの流れ

LA-920に採用した循環システム(図 7)は，測定フローセル部において分散媒中の粒子が常に均一に流れるように設計されている．遠心ポンプは2,000ミクロンの鋼球を含むサンプルの循環に対しても十分な能力を有する．

2 5 Accuracy Traceability

The pharmaceutical industry especially, among others, must be able to validate the performance of their instrumentation Their validation has to meet FDA requirements before any new process can be put on stream The LA-920 can easily be validated by measuring NIST-traceable standards, since the instruments are calibrated to these specifications before they leave the factory The result of a 1000-micron standard is shown in Figure 6

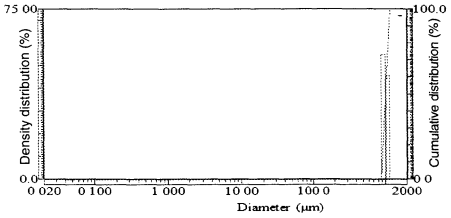

Fig 6 Measured result of 1000 μm standard

3. Dispersion Needs

Particulate systems occur in many different states of dispersion In some, each particle is discrete and separate Others may be weakly "stuck" together (flocculated or agglomerated) or strongly bonded (aggregated) And, depending on the application, some need to be measured in their naturally occurring state whereas others may need to be further dispersed, or separated from each other

3 1 Dispersing Energy

The LA-920 circulation system provides an in-stream 30-watt ultrasonic probe to allow the user to disperse the particles to any degree desired The probe can be used at a number of different power settings and for variable amounts of time The probe is located in the circulation stream in such a way that all the particles impinge on the probe tip, where the ultrasonic energy is at its maximum This means that particles can be completely dispersed in the minimum amount of time

3 2 Solvent Compatibility

All wetted surfaces of the circulation system are either stainless steel, titanium glass or Teflon With these inert materials, any of the liquids needed for carrying particles for size analysis can be employed with no problem

3 3 Representative Presentation

The LA-920 uses a newly designed centrifugal circulation pump and flow path to ensure that a representative sample of the size distribution appears in the sensing zone at all times The pump is powerful enough to circulate 2000-micron particles as heavy as steel, and the transition pieces from the circular Teflon tubing to the rectangular sample cell create a continuous flow distribution, free of any eddy currents, providing a representative sample across the entire sample cell The complete layout of the circulation system components and actual flow path are shown in Figure 7

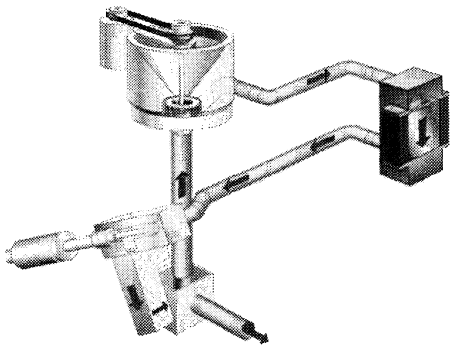

Fig 7 Newly developed circulation system

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4 自動化

LA-920のソフトウェアは，すべての操作をパーソナルコンピュータにより制御設定できるよう設計されている．最大24種の測定手順ファイルの組込みが可能で，個々のボタンやスイッチによる調整や操作をする必要がなく，自動的に測定手順を実行させることができる．

5 操作の容易性

5 1 データの取扱いと表示

Windows95™をベースにしたLA-920のソフトウエアは，測定結果の整理が容易にできる．粒度分析に使用される代表的なグラフおよび表の出力形式を網羅している．さらに，画面のレイアウトは，自由に設定することができ，異なったサンプルの測定結果を画面上の別々のグラフで表示できる(図８)．

4. Automation

Automation in analyzers offers two major benefits to the user: 1) it saves money by freeing up an operator's time, and 2) it gives consistently correct results by eliminating operator error and variability

The LA-920 sample delivery software gives the operator freedom to interact with the analyzer, perhaps when more in a research environment, or to set up automatic routines for on-going quality control applications

The LA-920 software provides keyboard control of the: 1) circulation pump and speed, 2) sample chamber fill pump, 3) sample drain actuation, 4) ultrasonic power and time, 5) an automatic dilution control function, and 6) measurement time The software also allows the operator to set up measure condition files where the sequence of sample preparation (e g , ultrasonic treatment) and delivery events are automatically executed As many as 24 different files of this type can be programmed for recall and use at any one time, but an unlimited number of groups of 24 can be stored for recall and use

5. Operational Conveniences

5 1 Data Manipulation and Display

The Windows based operational software for the LA-920 provides many user conveniences Data can easily be manipulated after the measurement to examine how the distribution would be reported with different optical parameters, or with a different number of calculation iterations The data can also be reported in a wide variety of graphical or tabular formats which cover all of the typical formats used in particle size analysis Furthermore, both the screen and hard copy report layouts can be formatted, independently, in the full variety of selections One screen layout contains numerous (depending on monitor type) separate sample reports displayed graphically for easy comparison purposes These data can then also be overlaid on one graph An example of this particular screen is shown in Figure 8

5 2 Data Retrieval and Batch Operations

A data storage system allows easy flexible retrieval of data records Data can be retrieved by various retrieval criteria The data, once retrieved, can be printed, exported to spreadsheets or other computers, or recalculated, in a batch operation, instead of individually, record by record

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5 2 データの修正･検索

ハードディスクなどに保存した測定結果を指定した基準に従って検索することができる．検索した結果をプリントし，再計算し，あるいは他のコンピュータに出力することができる

5 3 レポート書式のカスタム化

LA-920では，自社のタイトルやロゴなどを使って固有のレポートフォーマットを作成することができる．作成されたレポートの画面の例を示す(図９)．

5 4 機密保護

LA-920のソフトウエアは保護機能を持っている．保護の設定により，他の者が，測定条件のファイルなどに勝手に変更することが防止される．このときLA-920にアクセスするには，使用者が各自のパスワードを入力する必要がある．

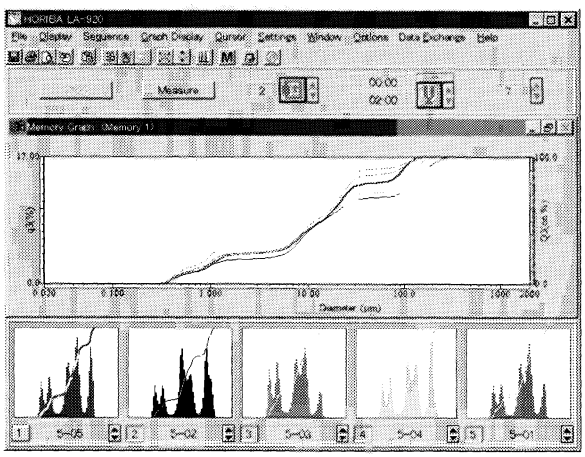

Fig 8 Screen display of multiple sample

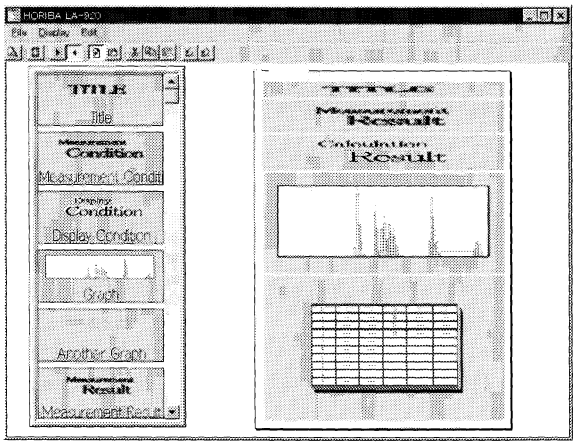

Fig 9 Screen display for creating reports

5 3 Custom Report Formats

Another convenience feature of the LA-920 is the ability to create custom report formats Figure 9 shows the screen for creating these reports Custom title blocks, logos and other company-specific formats can be created along with the specific report layout for the data

5 4 Security

A security function is also included in the LA-920 software This feature, required by most pharmaceutical companies and in an increasing number of other applications allows the lab supervisors to set up different levels of security These different levels prevent unauthorized personnel from changing such items as measure condition files, report display and hard copy formats, and other parameters that must be maintained All levels of access require passwords to be set up and entered by each user

6. Summary

The LA-920 comes with all the functions and features to meet the user needs in particle size measurement Performance is optimized; sample dispersion can be optimally achieved; automation is available in the operation of the instrument; and, finally, the analyzer software provides all the convenience features, from custom reporting to security, that are in demand today

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6 まとめ

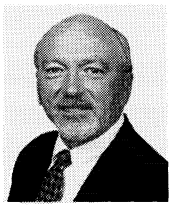
光散乱/回析法を使ったLA-920は，その測定性能，自動化，操作性などにおいて，最新のユーザーニーズにお応えできる，最も有用な粒度分布測定装置だと自負している

(抄訳 編集部)

All of these contribute to angular light scattering, and the LA-920 in particular, to being the most versatile particle size measuring system available - one that can meet almost any application

<Reference>

1) T Allen, *Particle Size Measurement*, Chapman & Hall, London, Fourth Edition, 1990
2) M Kerker, "The Scattering of Light and Other Electromagnetic Radiation", Academic Press, New York, 1969
3) E Heidenreich & M Stintz, *Evaluation of Particle Analysis Data Regarding Reproducibility and Accuracy* "Particle Size Analysis", Edited by N G Stanley-Wood & R W Lines, Redwood Press Ltd , Wiltshire, England, 1992



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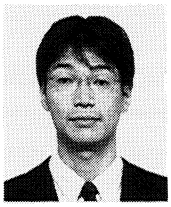

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