Total Solutions in Particle Characterization

APPLICATION NOTE

Blue Laser Technology Applied to the Microtrac Unified Scatter Technique for Full-Range Particle Size Measurement.

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Application Note

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Provided By:

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Particle Size Measuring Instruments

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INTRODUCTION

More than 3 decades ago, Leeds & Northrup developed the first truly commercial light scattering instrument [1] in the U. S. It was based solely on Fraunhofer diffraction and measured particle sizes in the range of about 2 to 200 microns. Several experimental laboratory and prototype units were in existence at that time, but they were generally manually operated devices with very limited capability [3,4]. In the late 1970's and early 1980's, L&N development teams extended the measurement range toward smaller sizes utilizing Mie scatter theory as well as differential polarization techniques to extend the size range down to 0.1 micron. This technology was state of the art at the time and filled many particle-sizing needs during the 1980's.

L&N development teams introduced during the 1990's a new "Unified Scatter Technique". This replaced the older differential polarization method with its associated problem of discontinuities when combining two different scatter methods into one integrated particle size distribution. This new method incorporated the use of both a forward and a high-angle detector array in conjunction with a new and unique TRI-LASER System, which effectively multiplies the number of logarithmic detectors available for scattered light detection. The three laser arrangement produced scattered light through an angular range from very close to zero degrees up to 160 degrees, in one continuous pattern.

The Tri-laser system has been upgraded to include blue lasers to provide advances in resolution and sensitivity in the submicron and nanosize ranges. The concept of utilizing logarithmic detector design to provide shift-invariance described for tri-laser systems is maintained and extended to the combined use of blue and near-infrared lasers

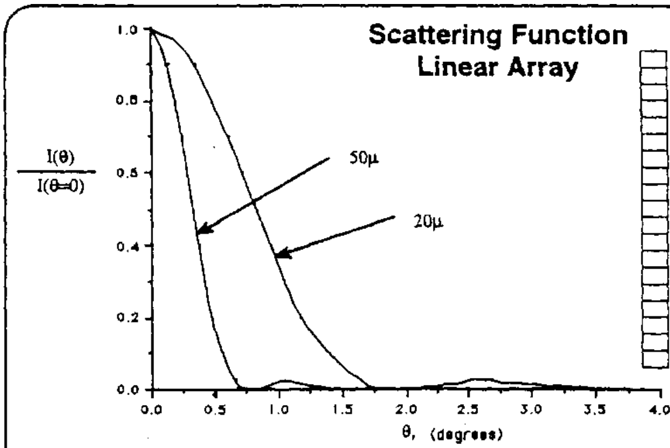
VOLUME RESPONSE

Light which is scattered by particles produces patterns that are proportional to the cross sectional area of the particles. Users of particle measurement systems usually desire results in terms of the "amount" of particulate material such as ***volume*** rather than area. Early MICROTRAC Analyzers employed a unique detector geometry that produces signals proportional to the volume of particulate material rather than area. This detector geometry has been utilized on all MICROTRAC Analyzer systems until the present day. The geometrical configuration is a rectangular line array of non-linear detectors, which converts scattered light flux into signals that are directly proportional to particle volume. These signals can be readily converted into a volume particle size distribution using a high-resolution iterative deconvolution algorithm.

LOGARITHMIC VS LINEAR ARRAY

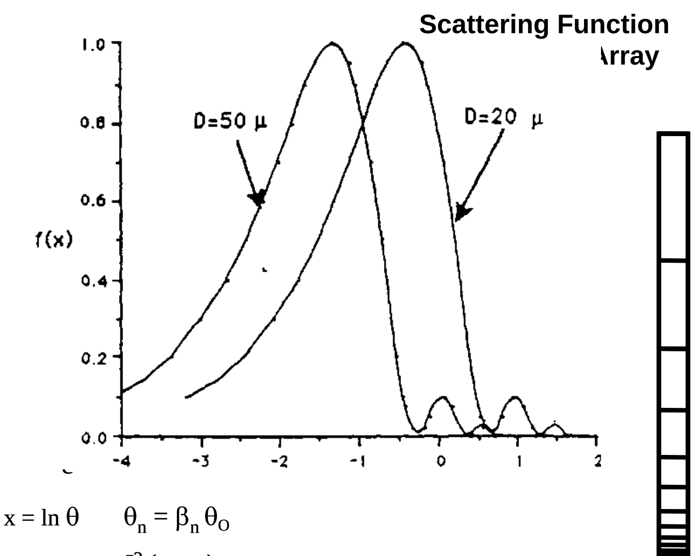
Scattered light, which is produced by particles in a laser beam, is not a linear phenomenon. In practical system designs, the low angle scatter (less than 10 or 15 degrees) can accommodate size ranges from hundreds of microns down to several microns. It takes very large angles beyond that to provide scattered light information for sizes below several microns down to 10 nanometers. For this reason, and also because scattered light intensities decrease with increasing angles, the detectors in a measurement system should get progressively larger as the scatter angle increases. That is why MICROTRAC systems have always utilized a logarithmic progression of detectors [5].

An advantage of the nonlinear detector array can be seen in the first 2 figures. The scattering functions and their plots show that the linear array (Figure 1) produces scatter intensities that are quite different for different sizes of particles (20 micron and 50 micron particles used as examples). In contrast, the logarithmic, array (Figure 2) produces patterns that are identical in shape for the different sizes, but simply displaced on a log-angle axis. This convenient shift-invariant functional relationship optimizes the data processing task of deconvolving a particle size distribution from scattered light flux.



$$I(\theta) = \frac{N E \lambda^2 \alpha^4}{16 \pi^2} \left(\frac{J_1(\alpha \theta)}{\alpha \theta} \right)^2 \qquad \alpha = \frac{\pi D}{\lambda}$$

Figure 1. Linear Line Array



$x = \ln \theta \qquad \theta_n = \beta_n \theta_O$

$$f(x) = KV \frac{J_1^2(e^{x-x_1})}{(e^{x-x_1})}$$

For volume distribution v9x0, $K_1 = 1$

$$f(x) = \int_{-\infty}^{+\infty} \frac{J_1^2(e^{x-x_1})}{(e^{x-x_1})} dx_1 = v(x)*h(x) \qquad hx = \frac{J_1^2(e^x)}{(e^x)}$$

Figure 2. Logarithmic Line Array

SINGLE LASER SYSTEMS

Instruments utilizing light scattering techniques employ many shapes and sizes of detectors- They range from small to very large and some take on very strange looking configurations. Some instruments use additional enhancements such as side scatter and polarization techniques. The one thing they all have in common is that they use ***one*** laser as the source.

Until the advent of the TRI-LASER System, MICROTRAC did the same, except for progressing from a gas laser to a solid state laser for long-term dependability and analyzer size reduction. A single laser with manipulation of detector geometry can produce real modal information in particle size distributions down to about 0.5 micron. If distributions are well-behaved, relatively wide, and have a single mode, the small particle response in single laser systems can be extended mathematically to approach the 0.1-micron limit that many systems claim.

However, if sample materials have components that contain modes below about 0.5 micron, the single laser systems cannot distinguish those modes from the main distribution or from other modes at larger sizes. More information is needed often in the form of blue LEDs, blue filters possibly in conjunction with polarization techniques. the disadvantages of which are discussed earlier, or a whole new approach which is provided in the multiple laser system discussed here.

THE TRI-LASER SYSTEM – Advanced use of blue and red/NIR lasers

An advanced TRI-LASER System has been developed by MICROTRAC which allows light scattering measurements to be made from the forward low angle region to almost the entire angular spectrum (approximately zero to 160 degrees). Until the advent of commercial lasers, “diffraction” measurements were difficult due to beam divergence, coherence, bandwidth and intensity. Similar issues are experienced with LEDs and filters. The original tri-laser system incorporated sources that used only laser technology. In the advanced system, use of blue lasers (shorter wavelength) provides distributions of enhanced resolution and sensitivity particularly in the submicron and nanosize regions of the particle spectrum. The application of blue laser technology in concert with advanced mathematical analysis enhances the ability of instruments to measure multi-modal and submicron particles with unsurpassed resolution and sensitivity. The primary laser (red/on-axis) produces scatter from nearly on-axis to about 60 degrees, utilizing a forward array and a high-angle array, both of which are logarithmic. The second laser (blue/off-axis) is positioned to produce scatter beyond the 60 degree level using the same detectors (see Figure 3). The third laser (blue/off-axis) is positioned to produce backscatter, again using the same detectors. This technique effectively multiplies the number of sensors that are available for detection of scattered light.

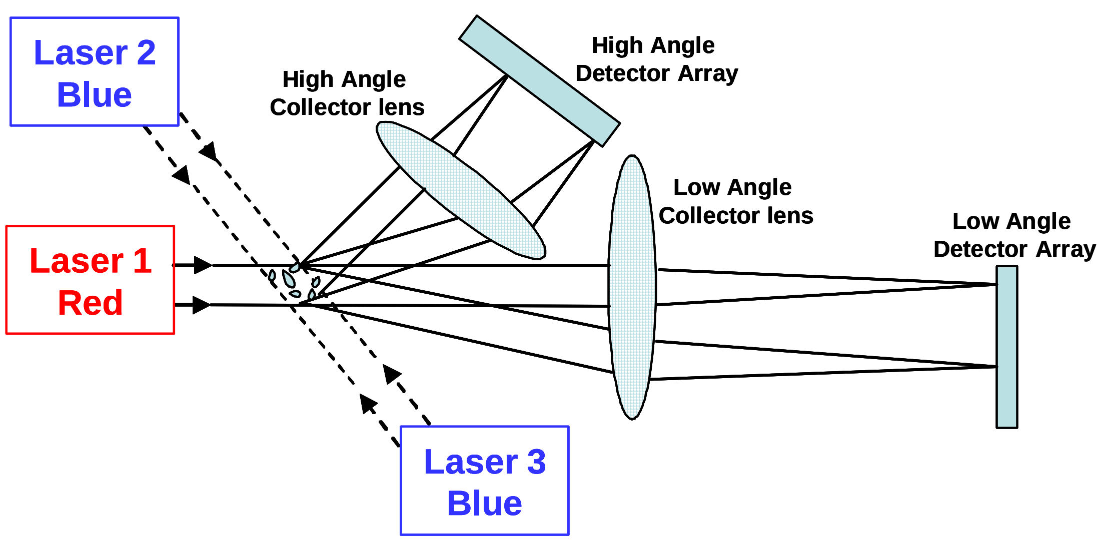


Figure 3. TRI-LASER System with Blue Lasers

As shown in Figure 4a, the laser can be motor-driven to various locations to change the incident angle for illuminating the particles. This movement, however, proves to be impractical due to the requirement of mechanical movement and the attendant issues of mechanical fatigue or failure. The lasers in the Tri-laser system are solidly placed at correct locations to change the incident angle of the beam. The use of a second laser in effect allows the wider angle scattering pattern to be re-located on the same detector. The use of a second laser and the same detector thus allows efficient use of one detector and 2 lasers. The Tri-laser system extends this concept by using 3 lasers and 2 detector arrays as shown in Figure 3.

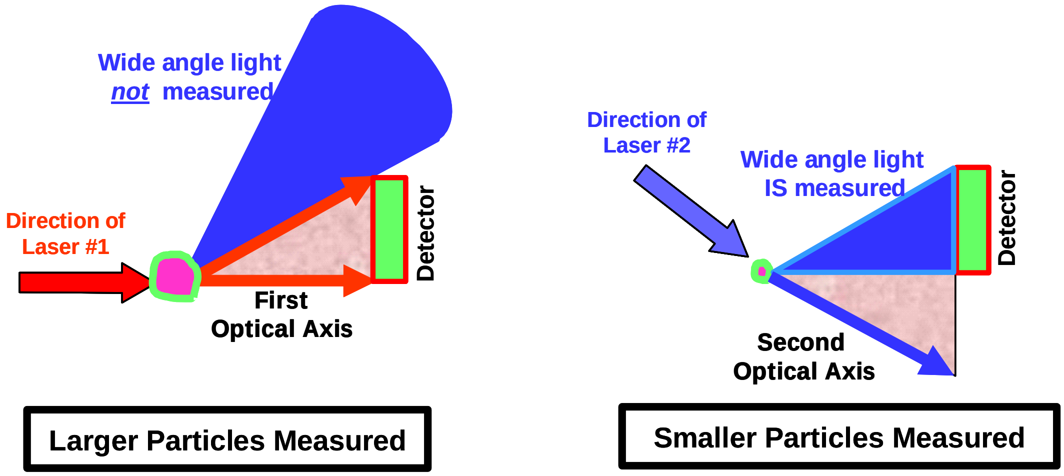


Figure 4a. Moving a single laser to new location or applying a second laser in a different location allows the same detector to be used to measure wider angles for smaller particles.

A better understanding of the extent of the angular range can be obtained by referring to Figure 4b. The on-axis detector collects a relatively small fraction of the total scattered light, but represents a large fraction of the total size range. Addition of an off-axis detector array markedly increases the angular range. The full TRI-LASER capability incorporating blue lasers increases the sensitivity at wide angular ranges tremendously, and provides a very important extension to full-capability response to submicron and nanosize partcles. It is this very large angular response combined with blue laser technology that provides the resolution capability
to readily separate individual modes down to the absolute limit of size response of the system.

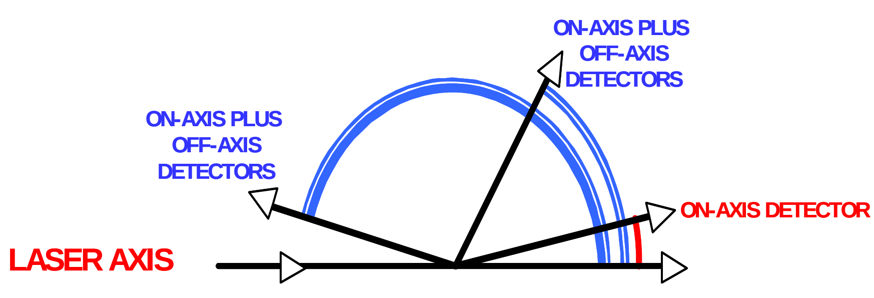


Figure 4b. Angular range of detection using 1 laser and multiple detector arrays. Near zero to 160º. The same can be accomplished using 3 lasers and limiting detector systems to 2.

DATA

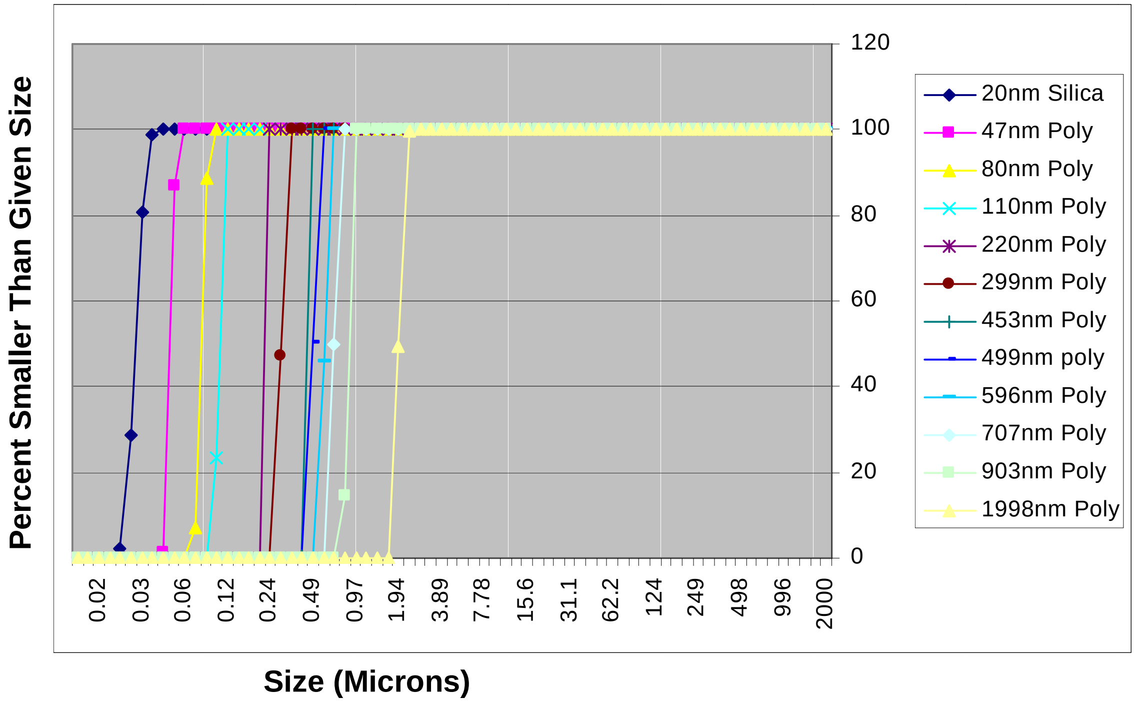


Figure shows comparison and capability of Bluewave to measure nano particles

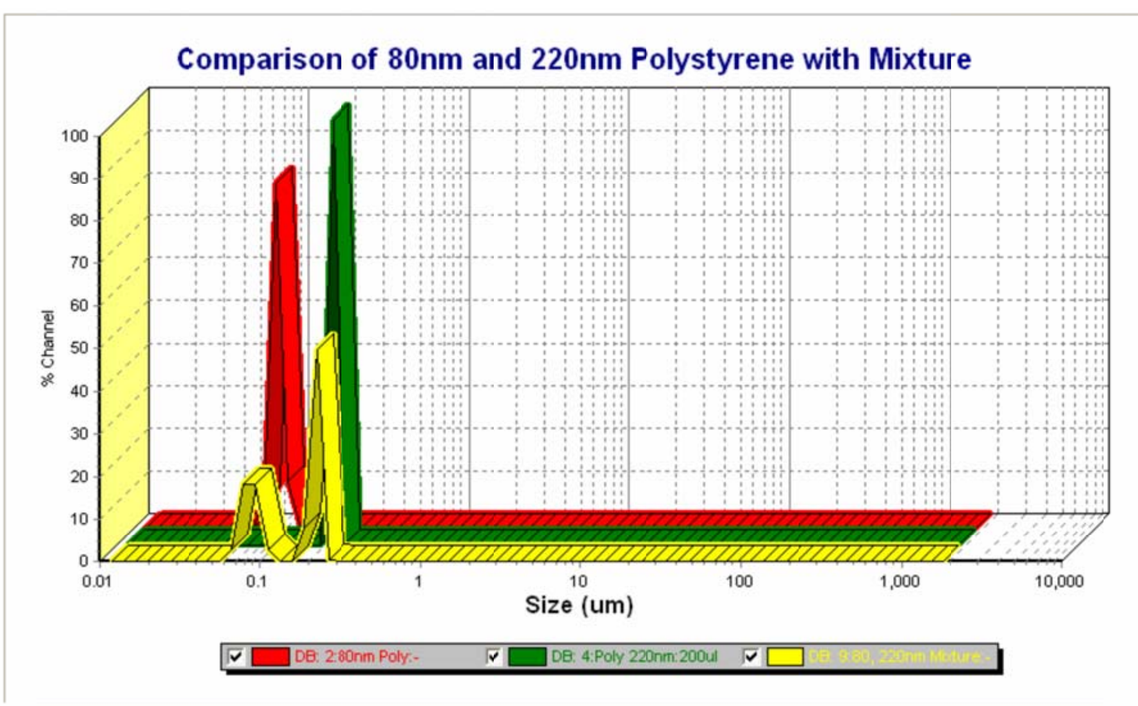


Figure shows a comparison of different size polystyrenes with a bimodal mixture of the two.
Note that size in mixture is same as individual polys

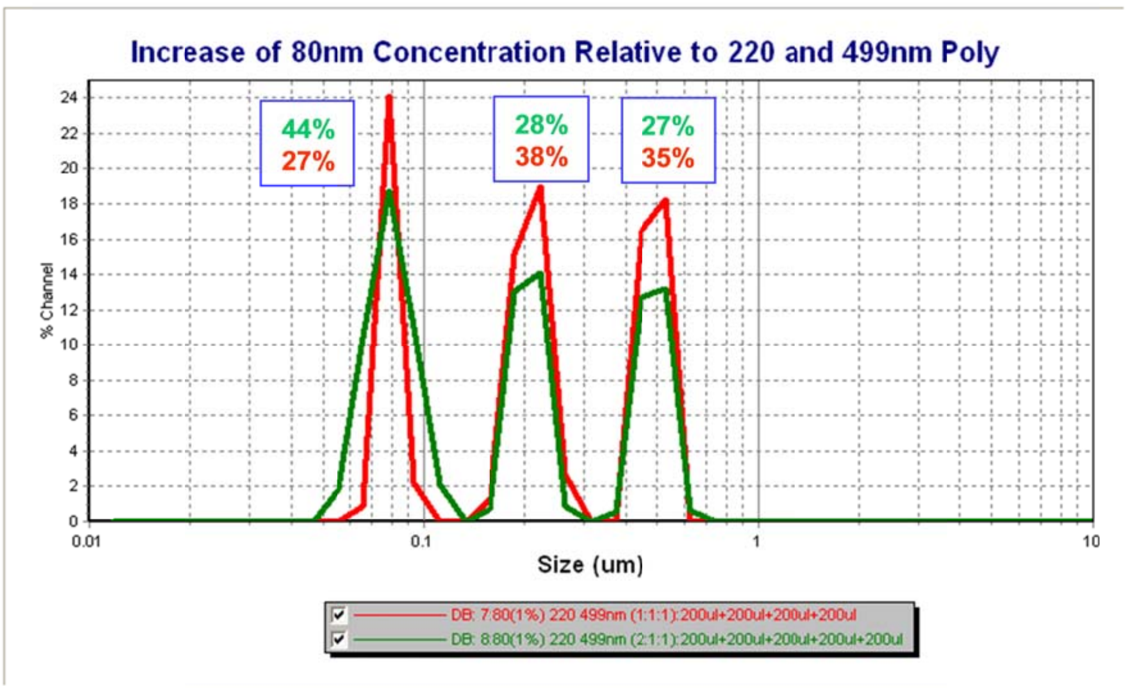


Figure shows that Bluewave is sensitive to changes of volume percent of nano size particles in a tri-modal particle size distribution

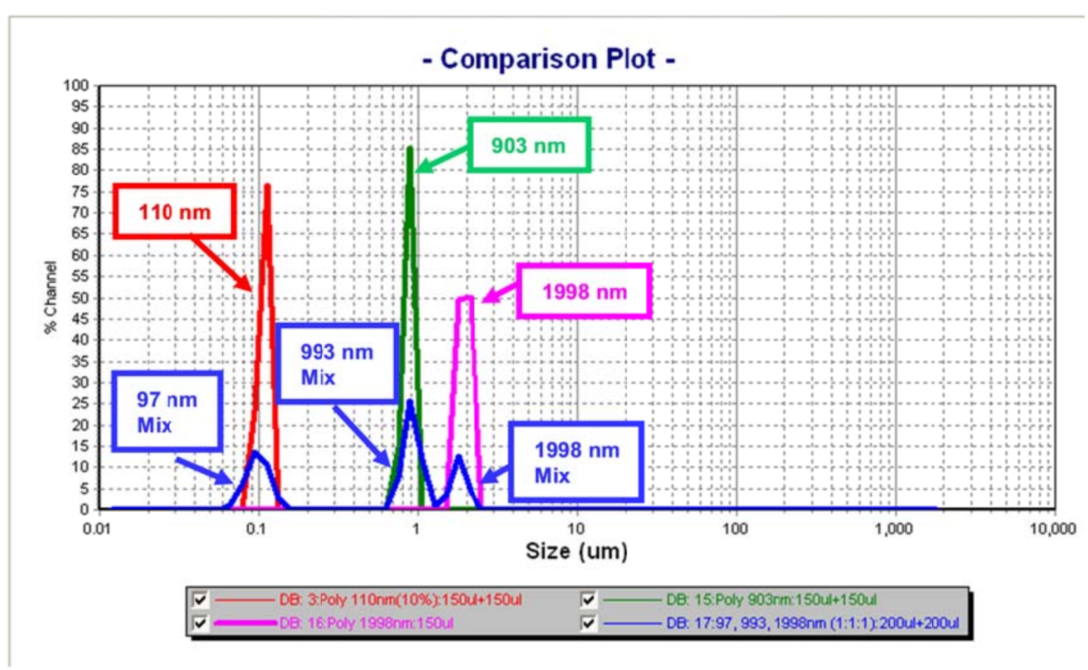


Figure shows size sensitivity of particles in a mixture compared to individual certified polystyrene samples.

SUMMARY

The foregoing discourse describes an advanced TRI-LASER particle size analysis system design (Microtrac BLUEWAVE) that is capable of measuring particle size down to 0.010 microns and as large as 2816 microns with no assumptions or curve fitting of the distribution. Blue lasers in combination with advanced mathematical analysis is capable of readily discerning individual modes throughout the submicron region by means of a "Unified Scatter Technique" without resorting to more than one scatter mechanism such as differential polarization. This unified technique utilizes a continuous pattern of scattered light flux from near forward to near backward components with no discontinuities.

These Bluewave instruments have been developed to enhance the precision of wide range “diffraction” particle size measurement, especially in the submicron and nanosize region. For very fine particulate sample materials, including biotechnology samples and those requiring no dilution, the choice should be the Nanotrac Analyzer with its measuring range of 0.8 nanometers to 6.5 microns using the principle of heterodyne Dynamic Light Scattering [6]. The instrument is capable of measurements using the unique “Dip–n-Run” particle size technique.

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