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Size Distribution by Light Scattering from Individual Particles*

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The particle size distribution of a polystyrene latex has been determined using a new light-scattering photometer which measures the scattered radiance as a function of scattering angle of single aerosolized particles as they are levitated in a laser beam. The results are in agreement with those obtained by conventional light scattering and by electron microscopy. In addition to the main population, two classes of smaller particles were observed. This single particle light-scattering technique offers the possibility of analyzing broader size distributions than heretofore amenable to light scattering and has the added advantage of not requiring any *a priori* assumptions about the form of the particle size distribution.

INTRODUCTION

The determination of colloidal particle size distribution is still fraught with difficulty and uncertainty. There are two aspects to this matter — the direct problem and the inverse problem. The direct problem is to calculate a measurable property of a well-defined system using known theory when the particle size distribution is known. With high-speed computers, this problem is simple. The inverse problem is to infer the particle size distribution from measured properties. This may be quite elusive even for the simplest cases and may often be intractable. Yet this is the goal of particle size determinations.

In this paper we will consider some advantages of a novel light-scattering photometer in solving the inverse problem. This photometer measures the scattered radiance of single aerosolized particles as a function of scattering angle as they are levitated electrically in a laser beam. This single particle light-scattering technique offers the possibility of analyzing broader size distributions than heretofore amenable to light scattering and has the added advantage of not requiring any *a priori* assumptions about the form of the particle size distribution. We will restrict our considerations to isotropic, homogeneous spheres.

Particle Size by Light Scattering

When the radii are less than about one-tenth of the wavelength, Rayleigh scattering theory can be used¹

$$R_{\Theta} = \frac{9}{2} \pi^2 \left(\frac{m^2 - 1}{m^2 + 1} \right)^2 \frac{NV^2}{\lambda^4} (1 + \cos^2 \Theta) \quad (1)$$

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where R_Θ is the Rayleigh ratio at scattering angle Θ , m is the relative refractive index, λ is the wavelength in the medium, N is the number of particles in a unit volume, and V is the volume of a single particle. The Rayleigh ratio is the energy scattered by a unit volume in the direction Θ , per steradian, for unit irradiance by unpolarized light. The first term gives the vertically polarized component of the scattered light and the second term gives the horizontally polarized component. Obviously, there is a very simple angular dependence of the scattered light. For Rayleigh scatterers, the Rayleigh ratio may be obtained either from scattering or transmissivity measurements.

In this case, the indirect problem is solved simply by calculation of the particle volume using Eq. (1). When the system is polydisperse, this yields the volume average value of the volume. Unfortunately, it is not possible to obtain additional moments of the size distribution from these results.

When the particle size becomes comparable to the wavelength, the angular dependence of the light scattering becomes quite complex and highly sensitive to particle size. This is illustrated in Figs. 1—4 where the angular gain for each of the polarized components, G_1 and G_2 , is plotted against scattering angle for various values of the size parameter $\alpha = 2\pi a/\lambda$ and for refractive index $m = 1.20$. The angular gain is related to the Rayleigh ratio by

$$G_u = \frac{G_1 + G_2}{2} = \frac{4R_\Theta}{Na^2} \quad (2)$$

For $\alpha = 1$, the scattering curves differ only slightly from those for Rayleigh scatterers for which the vertical component G_1 is independent of angle and

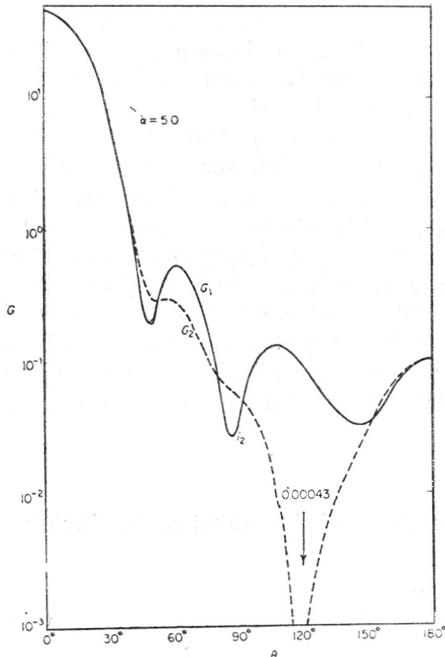


Fig. 1. Angular gain G_1 and G_2 for $\alpha = 1.0$ and 2.6 and for $m = 1.20$ plotted against Θ .

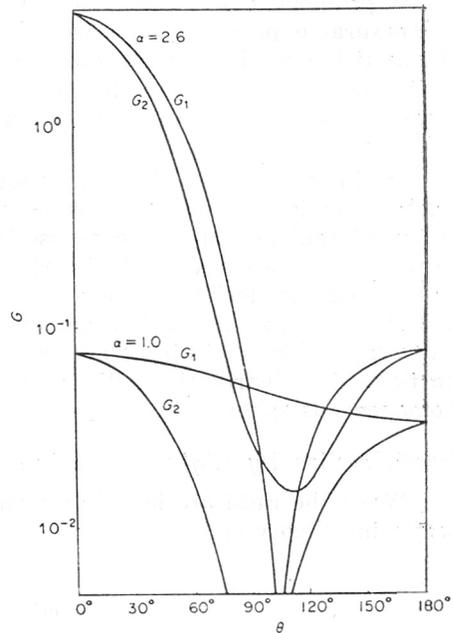


Fig. 2. Angular gain G_1 and G_2 for $\alpha = 1.0$ and 2.6 and for $m = 1.20$ plotted against Θ .

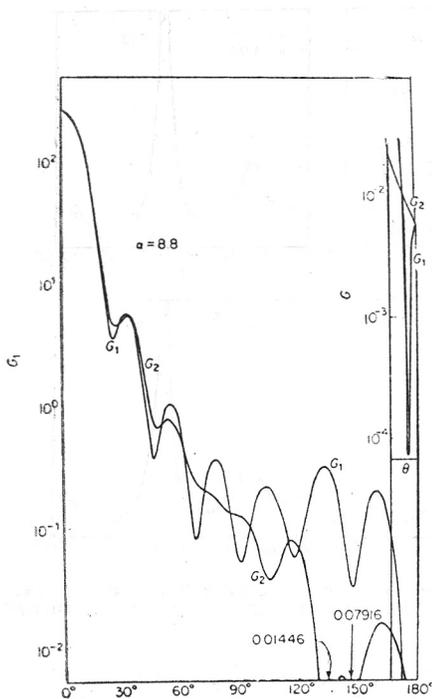


Fig. 3. Angular gain G_1 and G_2 for $\alpha = 8.8$ and for $m = 1.20$ plotted against θ .

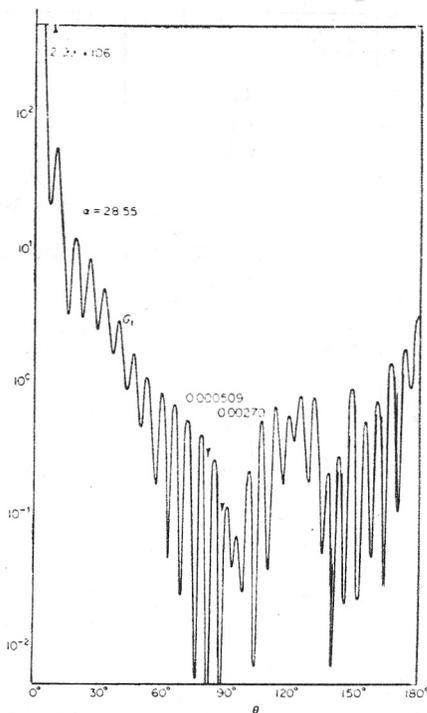


Fig. 4. Angular gain G_1 for $\alpha = 38.55$ and for $m = 1.20$ plotted against θ

the horizontal component, G_2 exhibits $\cos^2 \theta$ dependence. With increasing particle size, the pattern becomes increasingly oscillatory. The pattern also changes with refractive index so that it can serve as a »fingerprint« to determine both particle size and refractive index, thus solving the inverse problem.

There are experimental difficulties in obtaining suitable measurements from a single colloidal sphere. However, it has been possible to carry out appropriate measurements using a single silica fiber irradiated at perpendicular incidence². The physics of scattering by long circular cylinders is quite analogous to that by spheres, and so the potentialities can be appreciated if we consider the case illustrated in Fig. 5. The ordinate is the polarization ratio which is a convenient experimental parameter defined by

$$\rho = G_2/G_1 \tag{3}$$

This is plotted against θ , the scattering angle. The circles show the experimental results for a particular silica fiber with unpolarized light of 546 nm. These are compared with theoretical calculations shown as smooth curves for six combinations of the refractive index and the size parameter.

The results are striking. The best fit between the experiments and theory is for $m = 1.46$ and $\alpha = 4.00$ as depicted in the lower center panel of Fig. 5. When $\alpha = 3.98$ or 4.02 , the theoretical results differ from the experiments as shown. Similarly, there are differences between theory and experiment for $\alpha = 4.00$, but with $m = 1.45$ or 1.47 . In effect, as these diagrams show, this

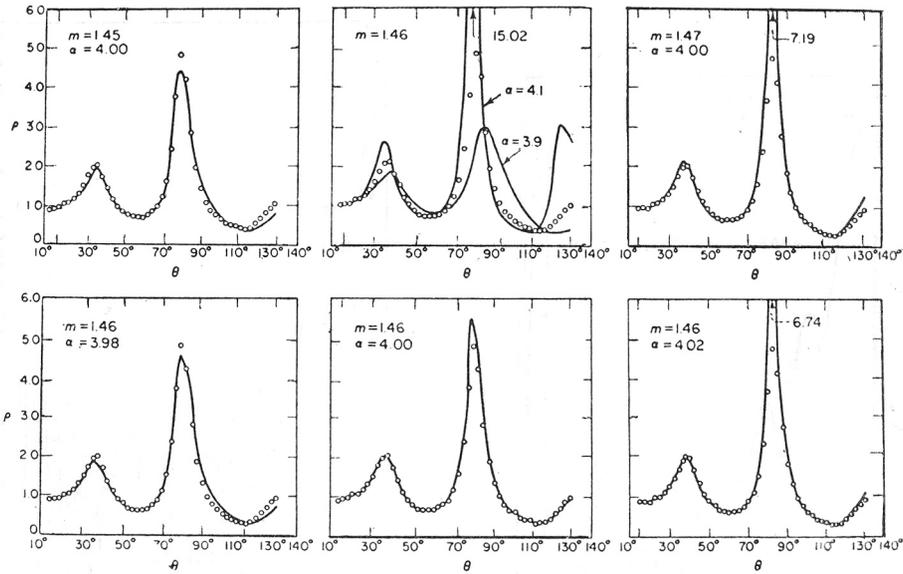


Fig. 5. Polarization ratio ($\rho = G_2/G_1$) plotted against θ for a Pyrex cylinder illuminated at perpendicular incidence. Comparison of experimental results (circles) and theory (smooth curves) for a silica fiber at $\lambda = 0.546$ nm.

represents a determination of the size of this fiber to an accuracy of about 0.25%. This is remarkably accurate. For example, size determination in this range by electron microscopy is hardly better than about 5%.

Single colloidal spheres cannot be manipulated easily so that in practice one encounters a dispersion for which the Rayleigh ratio must be integrated over the size distribution, wave length, and solid angle

$$R_{\Theta} = \frac{\int \int \int p(\alpha) R_{\Theta}(\alpha, \lambda, \omega) d\omega d\lambda d\alpha}{\int \int \int d\omega d\lambda} \tag{4}$$

where $p(\alpha) d\alpha$ is the fraction of particles in the size range α to $\alpha + d\alpha$ and $R_{\Theta}(\alpha, \lambda, \omega)$ is Rayleigh ratio corresponding to the appropriate values of α , λ , and ω . Light scattering photometers operate with monochromatic light, with parallel irradiation and with a small solid angle acceptance in the receiving system, and for these conditions the above equation reduces to

$$R_{\Theta} = \int p(\alpha) R_{\Theta}(\alpha) d\alpha \tag{5}$$

The solution of this equation for $p(\alpha)$ constitutes the indirect problem.

The effect of size distribution is illustrated in Fig. 6, which is based upon calculations for $m = 1.43$. The logarithmic distribution is characterized by the modal value of the size parameter (in this case $\alpha_M = 5.0$) and a breadth parameter which varies from $\sigma_0 = 0.100$ to 0.300. The distribution curves for the two extreme cases are in the inset, where the radii plotted along the abscissa correspond to light of wavelength 546 nm.

For $\sigma_0 = 0.100$, the curve shows the typical oscillations characteristic of a narrow distribution. However, this structured character is obliterated as the distribution becomes broader. It is the structure in these curves, acting as a

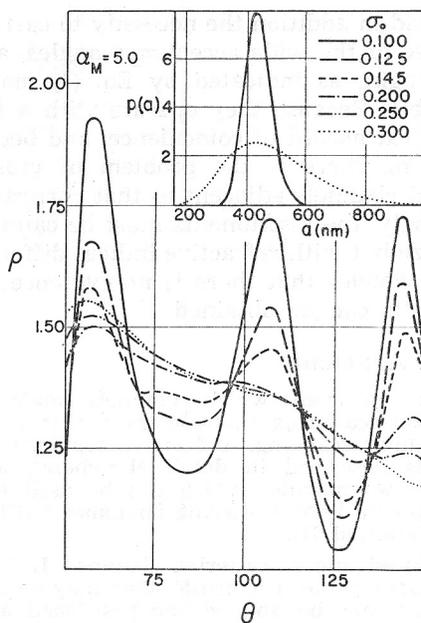


Fig. 6. Plot of $\rho(\theta)$ vs. θ for $\alpha_M = 5.0$ and $\sigma_0 = 0.100, 0.125, 0.145, 0.200, 0.250,$ and 0.300 . The inset show the corresponding extreme distribution curves. $a =$ radius in $m\mu$.

kind of fingerprint, that permits the precise determination of the size distribution from the light-scattering data. As the distribution becomes increasingly broader, it becomes increasingly difficult to obtain a unique solution to Eq. (4). This is the dilemma. In most cases, narrowly dispersed colloidal suspensions are highly contrived laboratory preparations. Most systems encountered both in the laboratory and in nature are too polydisperse for particle size analysis by light scattering or by any other technique which depends upon inversion of an equation such as Eq. (4). Wallace and Kratochvil³ have suggested $\sigma_0 = 0.20$ as an upper limit.

Optical Particle Counters

Techniques based upon single particle counting such as the optical and electron microscopes avoid the above difficulty, but then other problems arise. If light scattering could be applied to single particle counting, it could have the advantage of being highly accurate and non-destructive. Unfortunately, typical particle counters which have been available until recently sacrifice the design features necessary to obtain the appropriate information for accurate analysis. Indeed, although they operate on a light-scattering principle, they hardly utilize any of the potential of light scattering for particle size analysis. The aerosol particles flow through a small sensing zone where they are illuminated, one particle at a time. The scattered light gives rise to a photoelectric pulse which is classified and counted by a pulse height analyzer. The various commercial devices⁴⁻⁶ are fixed angle instruments with very wide-angle illuminating cones and collecting cones. Furthermore, they use white light. Accordingly, they sacrifice the critical information which comes

from an angular scan and in addition the necessity to carry out the integrations over solid angle because of the wide acceptance angles, and over wave length because of the white light, as indicated by Eq. (4) makes interpretation of the data quite impossible. Because they operate with a flowing system, there is the additional statistical hazard of coincidence, and because the illumination is not perfectly uniform, there is the problem of cross-channel sensitivity (counting of particles in channels adjacent to that corresponding to the correct particle size)⁷. Accordingly, the instruments must be calibrated, usually against particles of »known« size but with refractive indices differing from the material being studied. It is no wonder that there is no evidence to date that accurate or even consistent results can be obtained.

The Differential II Instrument

Ideally, we seek an instrument which combines single particle counting with the design features of light-scattering photometers, *viz.*, irradiance with parallel and monochromatic light, angular scanning, and low acceptance angle. The Differential II instrument*, which is described in detail elsewhere⁸, appears to meet these requirements. There are two modules which can be used with the instrument to convert it from a conventional light-scattering photometer (Differential I) to a single particle instrument (Differential II).

An aerosol is introduced into a scattering chamber. If the particles to be investigated are originally suspended as a hydrosol, they may be aerosolized by a nebulizer. An individual particle may be isolated and positioned with a laser beam by a combination of pneumatic and electrostatic controls. Once it is captured, the particle is »frozen« in this position indefinitely by an automatic servomechanism. The scattered light is picked up by a photo-detector which generates a signal proportional to the radiance. The detector system permits observation of the scattered light to within three degrees of the beam in both forward- and back-scattered directions. Angular scanning may be carried out manually or automatically at any of several scanning speeds and over variable ranges of angle. The read-out system which we utilized was a Houston Instrument series 3000 strip chart recorder. An argon-ion laser tuned to $\lambda = 514$ nm was the light source. The light was polarized with the electric vector perpendicular to the scattering plane.

In this study we have used the Differential II to compare the size distribution of a sample of polystyrene latex, obtained particle by particle, with that determined conventionally using the liquid suspension, as well as by electron microscopy. It should be noted that users of optical counters treat such polystyrene latexes as »monodisperse« because the size distribution is far narrower than these instruments can resolve.

The polystyrene latex produced by the Dow Chemical Company and designated as LS-449-E was reported by Dow to have a diameter of 796 nm with a standard deviation of 8.3 nm. A stock solution was prepared by diluting a portion of this sample 1000 to 1 with doubly distilled water. A drop of Vitan soap solution was added to stabilize the suspension.

Two to ten mls of this suspension were placed in the nebulizer of the Differential II light-scattering photometer and the nebulizer activated using nitrogen at 10–12 lbs pressure. A particle was then caught and suspended in the laser beam and a light-scattering run made by scanning from 60° to 155°. The particle was expelled from the light-scattering chamber with nitrogen and the procedure repeated. Values proportional to the scattered radiances were read from the recorder tracing at 5° intervals between 60° and 155°, and these were punched onto IBM cards for computer analysis.

It should be noted that occasionally a particle was captured which appeared very bright and which blinked. In such cases the recorder traces were quite irregular. We assumed that these particles were rotating doublets or multiplets, and these results were not utilized in this study.

* Manufactured by Science Spectrum, P. O. Box 303, Santa Barbara, California 93105.

Although there was some variation in brightness among the particles, there did not appear to be any apparent relation between the impression of brightness and the particle size, *i.e.*, upon analysis some of those particles which appeared dimmer turned out to be among the larger ones. There is the possibility, because of the variable appearance of the particles, that the observer might introduce a bias into the selection of those particles for capture. However, since visual observation was at a fixed angle and in a particle size regime where there did not appear to be any correlation between brightness and particles size, we attempted to reduce any such bias by capturing particles over a wide range of intensities.

For the light-scattering work on the aqueous dispersion, the Differential I module replaced the Differential II module converting the device from a single particle counter to an instrument which could be utilized in the conventional way. The background signal was monitored with the cell filled with 50 ml of doubly distilled water and then data were obtained with from 2 to 20 droplets of the sol added. The specific radiances were independent of concentration so that for this range it was not necessary to extrapolate to zero concentration. Both a conventional cylindrical cell with flat entrance window and a special tapered cylindrical cell provided with this instrument were used. The appropriate sine correction for the illuminated volume was made in each case.

Samples for electron microscopy were prepared by placing a drop of latex suspension (thousand-fold dilution) onto a colloidion-coated grid. This was allowed to dry and then placed directly into the Philips EM 100 electron microscope. The electron micrographs were analyzed with the aid of a Karl Zeiss particle counter.

Particle Size Analysis with the Differential II

Previously, particle size analysis had been carried out in this laboratory by comparison of measured values of the polarization ratio as a function of angle of observation with values calculated for various logarithmic distributions of spheres having the appropriate refractive index¹. The modal radius and breadth parameter which led to the best fit between experiment and calculation were selected to characterize the size distribution of the aerosol.

The polarization ratio is the ratio of the scattered radiances of the linear polarized components for which the electric vector is parallel and perpendicular, respectively, to the scattering plane. This ratio provides a scaling factor which eliminates the necessity of calibrating the instrument for absolute values of the radiances.

Our particular instrument only measured the perpendicularly polarized radiance and so, instead of utilizing the polarization ratio, the radiance was appropriately scaled by referring the measured signal at each angle to that at a reference angle as follows

$$S_{\Theta} = V_{\Theta} / V_R \quad (6)$$

where V_{Θ} is the perpendicularly polarized radiance at scattering angle Θ , and V_R is the value at the reference angle. The criterion for selecting the best fit between experiment and calculation was to minimize the deviation measure

$$E = \sum_{\Theta} (S_{\Theta} - S'_{\Theta})^2 \quad (7)$$

where S'_{Θ} represents the calculated value.

In the single particle work, calculations were carried out for $\alpha = 2.00$ to 8.00 in steps of 0.01. In the case of the dispersions, the modal value of the size parameter was varied from $\alpha_M = 4.00$ to 7.00 in steps of 0.01, and the breadth parameter of the logarithmic distribution varied from $\sigma_0 = 0.01$ to 0.10 in steps of 0.01. These results were analyzed with the aid of the error contour

plots described earlier¹. For the aerosol particle, the refractive index was 1.600 and that for the hydrosol was 1.202. Of course, for those experiments based on the observation of a single particle, S_θ is determined only by the value of the radius. For those experiments with dispersions, it depends upon the two parameters of the distribution, the modal value of the radius, and the breadth parameter.

There is a problem in selecting the reference angle. It was found that the radius obtained for the single particle, as well as the modal radius of the dispersions, depended somewhat upon the particular reference angle. Values obtained with reference angles near minima in the curve of radiance *versus* scattering angle deviated most sharply from the other values. Accordingly, the following procedure was used. Values of the radius were obtained using each of seventeen angles (60° [5°] 140°) as the reference angle. These were averaged and those values which differed from the average by more than twice the standard deviation were discarded and a new average and standard deviation were calculated. This process was continued until there were no values outside of these limits. This average was selected as best characterizing the particular run.

The sensitivity of the radiance to particle size is illustrated in Fig. 7 in which R_θ is plotted against θ for $\alpha = 4.70$ and 5.00 for $m = 1.600$. The variation of the deviation measure for a typical run is shown in Fig. 8 where E is plotted against the size parameter α . In this case, $\alpha = 4.89$ was selected as the appropriate size.

The experimental data utilized in Fig. 8 were obtained with a particular particle which had been levitated in the instrument for a period of eight hours and forty-five minutes in order to determine the stability of the

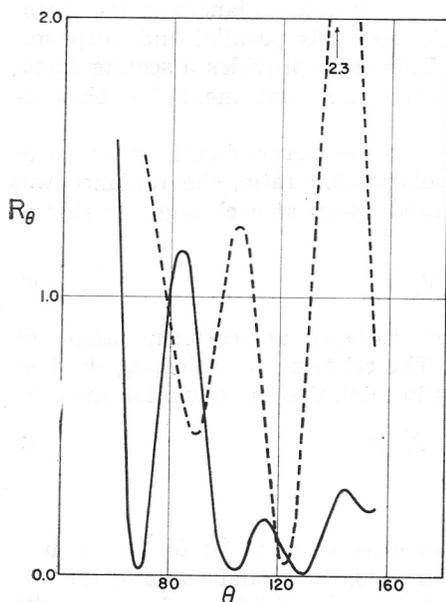


Fig. 7. Vertical component of radiance, R_θ , versus scattering and θ for $m = 1.600$; $\alpha = 4.70$ (dashed), $\alpha = 5.00$ (smooth).



Fig. 8. Deviation measure, E , versus size parameter, α , selected to fit a typical experimental run.

particle and the reproducibility of the data. The results for nine runs obtained over this period of time are shown in Table I. The standard deviation from the average value of $\alpha = 4.88$ is ± 0.02 . The last two columns in this Table illustrate the best fit to the experimental data when the calculations are carried out for α intervals of 0.005 and 0.001, respectively, rather than for intervals of 0.01. Obviously, there is no advantage to the shorter intervals. The variations in the size obtained over this period of time appear random and are probably due to errors associated with computational and instrumental uncertainties rather than to permanent changes in the particle such as might occur as a result of evaporation, condensation, etc. Similar experiments were performed with 16 other particles with comparable results.

TABLE I
Determination of the Size Parameter

Nine determinations of the size parameter α upon a single particle levitated in the laser beam for a period of eight hours and fortyfive minutes.

| Run No. | $\Delta \alpha$ step 0.01 | $\Delta \alpha$ step 0.005 | $\Delta \alpha$ step 0.001 |
|---------|------------------------------|-------------------------------|-------------------------------|
| P 1-1 | 4.89 | 4.890 | 4.892 |
| P 1-2 | 4.90 | 4.895 | 4.896 |
| P 1-3 | 4.85 | 4.855 | 4.853 |
| P 1-4 | 4.88 | 4.875 | 4.877 |
| P 1-5 | 4.88 | 4.885 | 4.884 |
| P 1-6 | 4.84 | 4.845 | 4.843 |
| P 1-7 | 4.90 | 4.905 | 4.904 |
| P 1-8 | 4.88 | 4.880 | 4.880 |
| P 1-9 | 4.92 | 4.915 | 4.915 |

Some error is introduced in the process of reading the values of the radiance from the recorder traces. These values are then punched onto cards for insertion in the computer. When the same data are read successively by both the same individual and various individuals, one obtains a standard deviation of about 0.01 in the value of α . There is equipment, which we do not possess, which provides the output digitally and which can be interfaced directly with a card punch. This would reduce this human aspect of the experimental error.

RESULTS AND DISCUSSION

The size distribution made up of 331 single particle runs is shown in Fig. 9 as a histogram of percent frequency *versus* size parameter α . The maximum frequency is at $\alpha = 0.79$ with 90% of the particles between 4.58 and 4.90. The confidence in these values is quite good as evidenced by low values of the deviation measure. The remaining particles are grouped discretely in the ranges $\alpha = 3.0$ to 3.3 and $\alpha = 3.72$ to 4.32, and for these the deviation measure is somewhat higher.

The average size of the major population group is $\alpha = 4.79$ or a diameter $d = 784$ nm. If the two smaller population groups are averaged in, this gives

$\alpha = 4.67$ or $d = 764$ nm. The value reported by Kratochvil and Wallace⁹ based upon light scattering from the latex hydrosol is 773 nm with a breadth parameter $\sigma_0 = 0.05$.

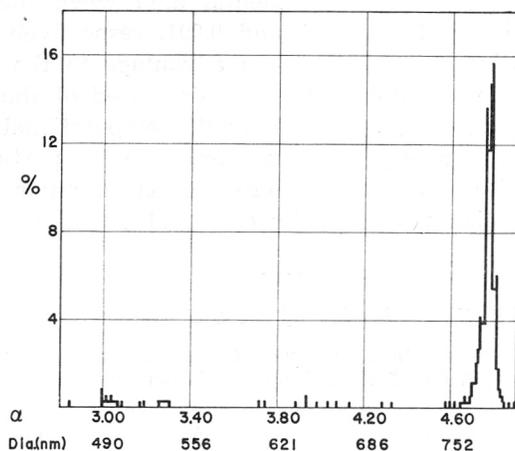


Fig. 9. Histogram of particle sizes obtained by light scattering for 331 particles.

The histogram obtained by counting 715 particles on the electron micrographs is shown in Fig. 10. In this case, we have calibrated the electron micrographs against the Dow study so that the peak size corresponds to the average size cited by Dow. The purpose here is to illustrate the presence of the smaller classes of particles. These do not correspond quantitatively to the single particle light-scattering results, yet they demonstrate the presence of such particles.

We have also carried out eleven separate light-scattering runs on the hydrosol using the Differential I instrument and have obtained an average modal diameter of 797 nm with a breadth parameter $\sigma_0 = 0.045$. Values for

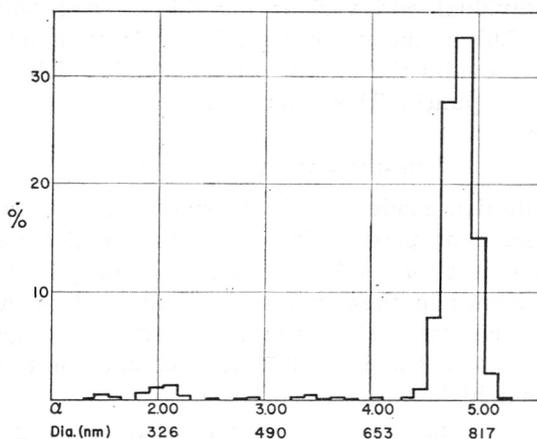


Fig. 10. Histogram of particle sizes obtained by electron microscopy for 715 particles.

the individual runs are shown in Table II. The light-scattering values compare with $d = 796$ nm reported by Dow using electron microscopy.

TABLE II

Size Distribution Obtained with a Hydrosol Sample using the Differential I Technique

| Run No. | α_M | σ_0 | d_M |
|---------|------------|------------|-------|
| 1-1 | 6.45 | .04 | 0.792 |
| 2-1 | 6.48 | .04 | 0.795 |
| 2-2 | 6.48 | .04 | 0.795 |
| 2-9-1 | 6.48 | .05 | 0.795 |
| 3-4 | 6.48 | .05 | 0.795 |
| 3-5 | 6.48 | .05 | 0.795 |
| 3-6 | 6.51 | .04 | 0.799 |
| 3-7 | 6.53 | .04 | 0.801 |
| 3-8 | 6.51 | .05 | 0.799 |
| 3-9 | 6.51 | .05 | 0.799 |
| 3-10 | 6.52 | .05 | 0.800 |
| Average | 6.49 | .045 | 0.797 |

The two smaller populations have not been reported before. It is not surprising that they would not be detected by conventional light scattering. For example, we have calculated the light-scattering radiances that would be obtained for the distribution depicted in Fig. 9 and then have inverted these results on the assumption that the system was a logarithmic distribution. This gave a modal size $\alpha_M = 4.75$ instead of 4.90 and $\sigma_0 = 0.02$. Thus, the smaller class of particles affects the estimate only slightly, and, if there were not some *a priori* information concerning them, there would be no reason, on the basis of the light-scattering data, to suspect their presence. Accordingly, light scattering from the assembly of particles cannot be used to detect the presence of these smaller particles.

The results are summarized in Table III, where values obtained for this latex by others are also cited. The differences are probably within the experimental errors of each of the techniques. The principal advantage of the single particle techniques (both light scattering and electron microscopy) is that no

TABLE III

Comparison of Average (or modal) Diameters of Dow Polystyrene Latex

| M e t h o d | Diameter nm |
|--|----------------|
| Particle by particle light scattering (this work) | |
| a. Major population group | 784 |
| b) All particles | 764 |
| Hydrosol light scattering (this work) | 797 |
| Hydrosol light scattering, Kratochvil and Wallace ⁹ | 773 |
| Electron microscopy, Dow Chemical Co. | 796 |
| Electron microscopy, Robbins and Jizmagian ¹⁰ | 777 |
| Electron microscopy, Davidson <i>et al.</i> ¹¹ | 765 |
| Electron microscopy, Cooper and Parfitt ¹² | 850 |
| Flow ultramicroscopy, Davidson <i>et al.</i> ¹¹ | 849 |

a priori assumptions are required regarding the form of the distribution function. Indeed, the negative skew of the main population group in Fig. 9 and the presence of the two classes of smaller particles could not be predicted nor can their presence be confirmed using conventional light-scattering techniques.

The failure heretofore to observe the smaller particles calls for further investigation, using both light scattering and electron microscopy. Also, much work remains to be done to assure the lack of bias in the sampling procedure for the single particle light scattering; otherwise, the question of the skewness of the distribution cannot be resolved definitively. In any case, the prospect for applying light scattering to size distributions broader than any heretofore amenable to light-scattering analysis appears to be a reasonable prospect.

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IZVOD

Određivanje raspodjele veličine čestica mjerenjem raspršenja svjetlosti na pojedinačnim česticama

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Određivanje funkcije raspodjele veličine čestica koloidnih disperzija još uvijek je opterećeno poteškoćama i nesigurnostima. Ovaj rad opisuje određivanje raspodjele veličine čestica, iz mjerljivih svojstava, primjenom novog tipa fotometra za promatranje, pozicioniranje i mjerenje raspršenja svjetlosti laserskog snopa na jednoj jedinjoj lebdećoj čestici. Čestica se odabire i fiksira u laserskom snopu pomoću kombinirane automatske pneumatske i elektrostatske kontrole. Dobiveni rezultati u skladu su s onima, koji se dobivaju za isti materijal polistirenskog lateksa klasičnom metodom mjerenja raspršenja svjetlosti ili pomoću elektronske mikroskopije. Osim što su mnogo širi rasponi veličina čestica pristupačni mjerenjima, ova tehnika ima prednosti i u tome, što ne zahtjeva nikakve, *a priori*, pretpostavke o funkciji raspodjele.

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