

Particle Characterization Laboratories, Inc.

Analytical services

Particle size analysis

- Dynamic Light Scattering
- Static Light Scattering
- Sedimentation
- Diffraction

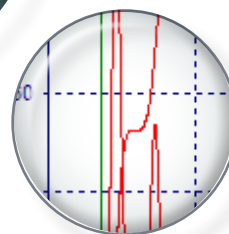
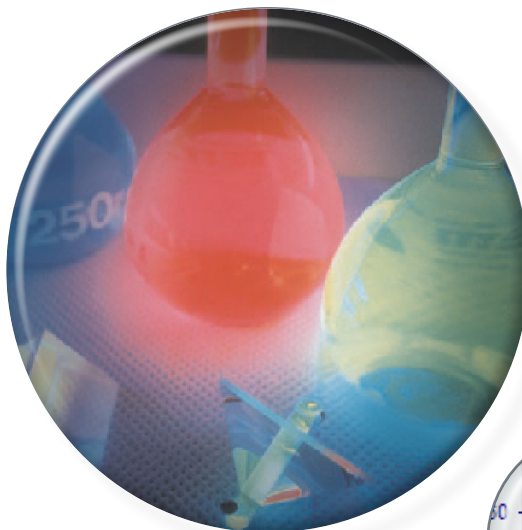
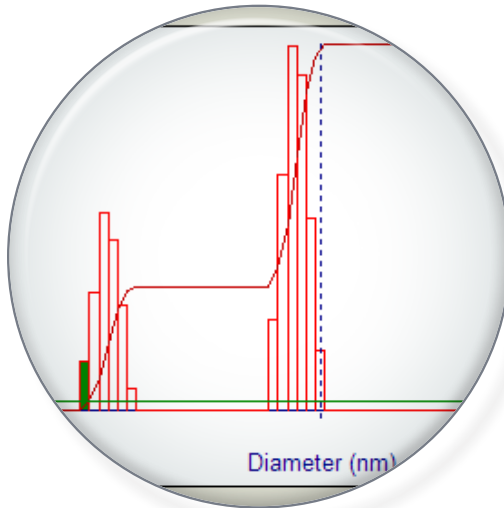
Zeta Potential Analysis

- Single Point Titration
- Iso-electric point determination
- Aqueous and non-aqueous media
- High Ionic
- Strength/Viscosity

Molecular Weight Analysis

- GPC Method
- Static Light Scattering
- Contrast Ratio (dn/dc)

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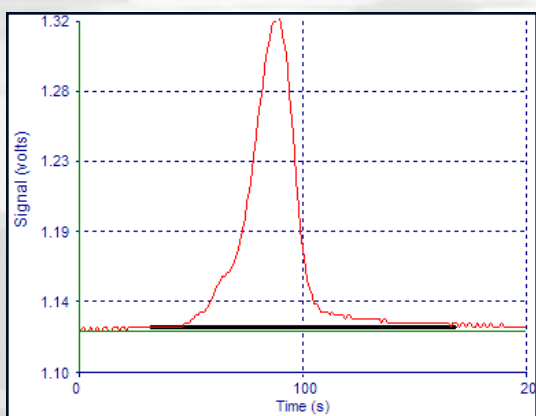


Particle Sizing

Particle Sizing

The overwhelming majority of natural and manufactured products involve particulate materials either in their final state or at some stage in their production. Particle size and particle size distribution are, therefore, often critically important parameters.

At the simplest level, information on particle size can help maintain a more consistent product, enhancing end-use value and profitability. At a more complex level, careful control of particle size can reduce the need for in-process modifications and reworking, so making products more competitive. There are many effective ways to determine the size and distribution of a colloidal dispersion.



Standard Sedimentation graph

$$t = \frac{18 \eta \ln \left(\frac{R_d}{R_j} \right)}{\omega^2 d^2 \Delta \rho}$$

Stokes Sedimentation Equation

Sedimentation

While the disc is spinning, a small volume (typically 1/4 mL) of a dilute suspension (typically less than 1 % concentration) is injected onto the surface of a liquid (spin fluid) previously injected into the disc cavity. The particles sediment radially through the fluid in the centrifugal field. Spin fluid volumes are typically on the order of 15mL.

Based on the principle of photo sedimentation in a disc centrifuge, the photo disc centrifuge utilizes the buffered line start, external gradient line start or homogeneous start methods of operation. The sedimentation behavior of particles depends on the particle density and the density and viscosity of the liquid through which the particles sediment. The time, t , for a spherical particle of diameter, d , to travel from the initial radius, R_j , at the surface of the spin fluid to the radius at the detector, R_d .

Light from a narrow-band, LED is passed through the disc. As particles pass through the light beam, the light intensity is reduced due to scattering and absorption. The transmitted light is measured by a photodiode and recorded by the computer as a function of time.

Services

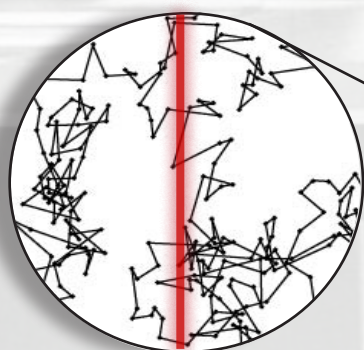
PCL offers high resolution results to particle sizing problems where the particles are dispersed in a liquid and cover the size range from 0.01 to 30 microns.

Material applications include:

- Polystyrene, PVC, other polymers
- Carbon blacks
- Metal oxides, refractories
- Ink particulates, alumina, titania
- Pharmaceuticals, cosmetics, foods
- Coatings, paints
- Minerals, silicas
- Clays, ceramics

Dynamic Light Scattering

The short-term fluctuations (dynamics) arise from the fact that the particles are small enough to undergo random thermal (Brownian) motion and the distance between them is therefore constantly varying. Constructive and destructive interference of light scattered by neighbouring particles within the illuminated zone gives rise to the intensity fluctuations at the detector plane which, as it arises from particle motion, contains information about this motion. Analysis of the time dependence of the intensity fluctuation can therefore yield the diffusion coefficient of the particles from which, via the Stokes Einstein equation, knowing the viscosity of the medium, the hydrodynamic radius or diameter of the particles can be calculated.



A diagram depicting Brownian diffusion

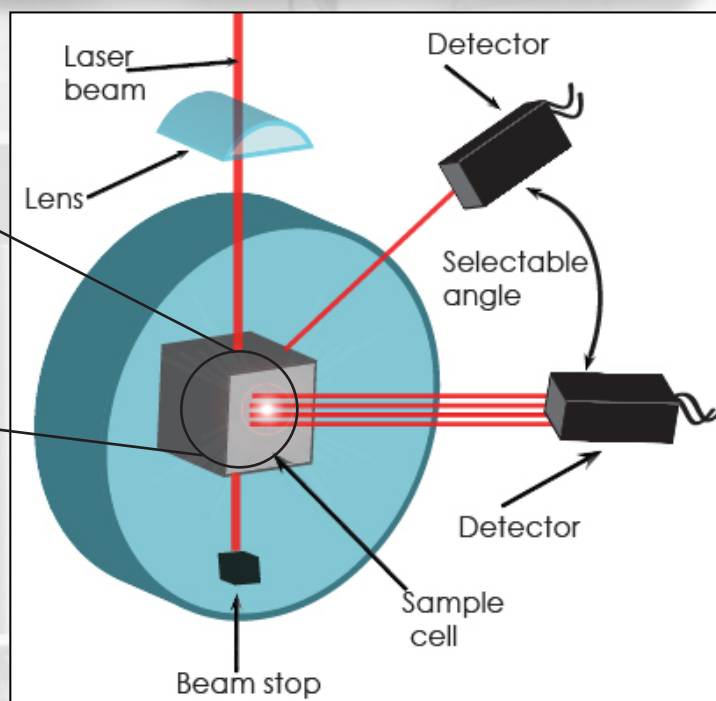
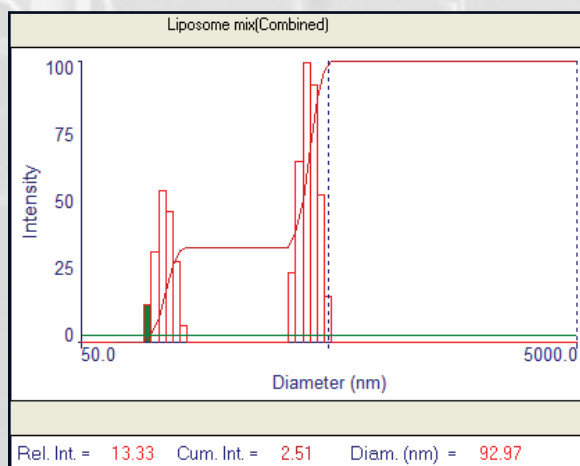


Diagram depicting a basic DLS apparatus.



Typical DLS output

Services

PCL offers the following DLS measurements:

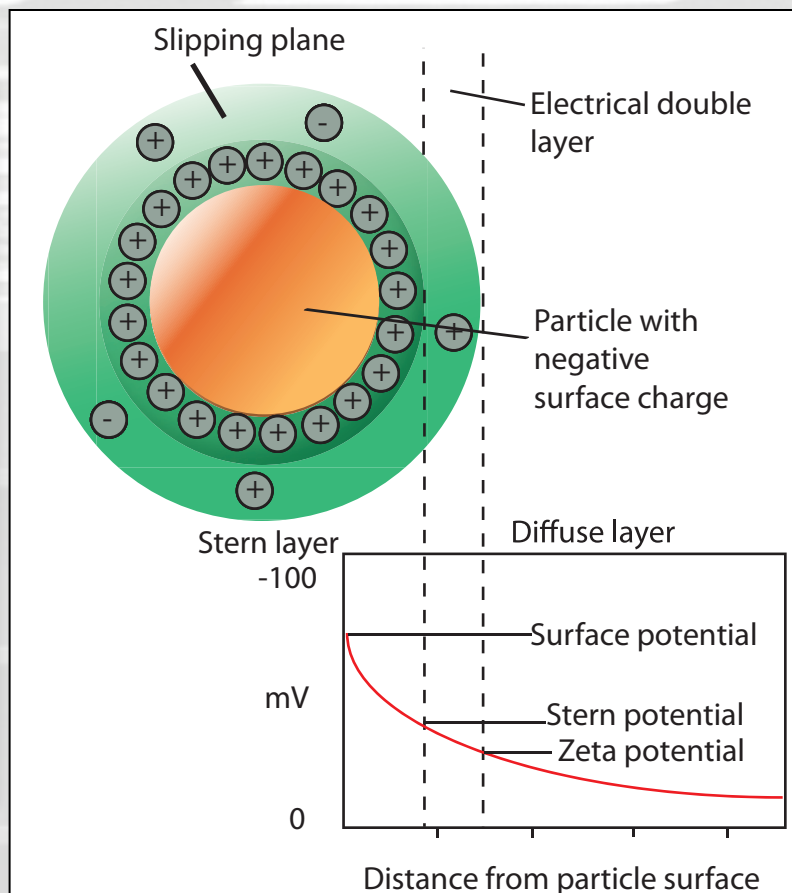
- Particle size distributions
- Particle aggregation rate
- Micellar systems
- Micro-emulsion technology
- Colloid behavior
- Vesicles & liposomes
- Plasmid DNA's
- Particle size growth
- Nucleation Processes & protein crystallization

Zeta Potential

Zeta Potential

Almost all particulate or macroscopic materials in contact with a liquid acquire an electronic charge on their surfaces. Zeta potential is an important and useful indicator of this charge which can be used to predict and control the stability of colloidal suspensions or emulsions. The greater the zeta potential the more likely the suspension is to be stable because the charged particles repel one another and thus overcome the natural tendency to aggregate. The measurement of zeta potential is often the key to understanding dispersion and aggregation processes in applications as diverse as water purification, ceramic slip casting and the formulation of paints, inks and cosmetics.

Zeta potential can also be a controlling parameter in processes such as *adhesion*, *surface coating*, *filtration*, *lubrication* and *corrosion*. Consequently, the presence or absence of charged groups on the surface of macroscopic materials such as hair, glass fiber, paper pulp, plastic films and refractories, as revealed by their zeta potentials can directly affect their performance and processing characteristics.

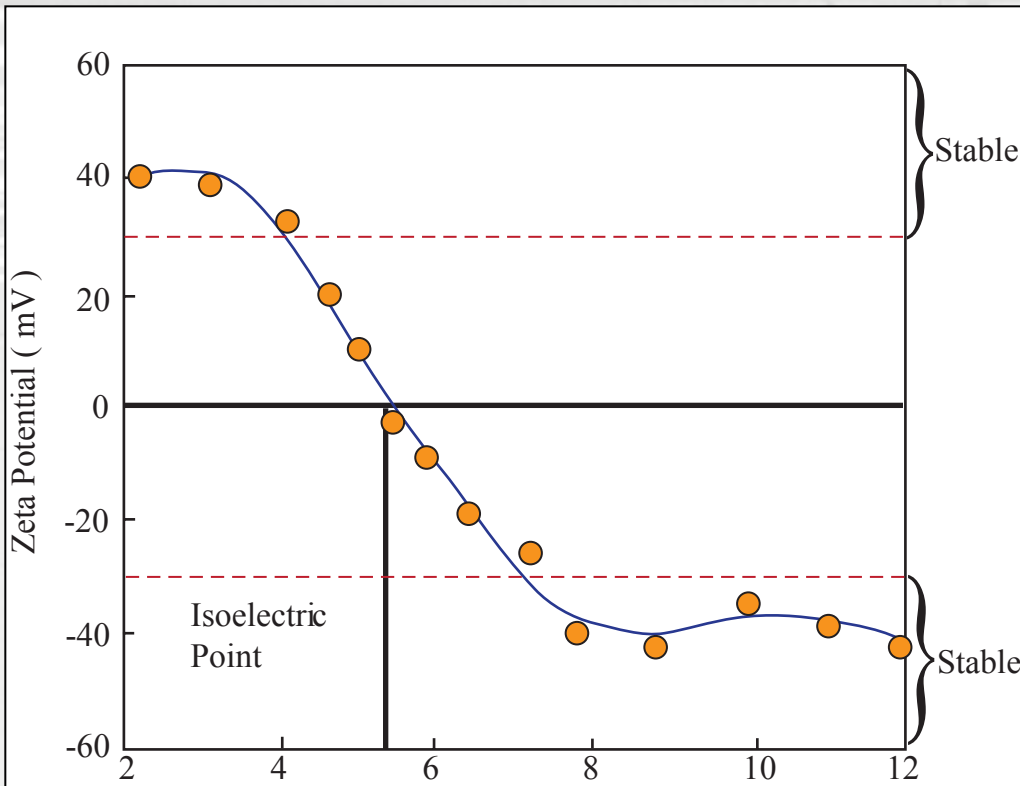


Candidates for Zeta Potential Analysis

- Liposomes and biocolloids
- Ceramics and refractories
- Pigments and inks
- Pharmaceuticals
- Emulsions (foodstuffs, cosmetics)
- Wastewater treatment monitoring
- Latexes
- Carbon blacks

Microelectrophoresis

The principal of determining zeta potential by microelectrophoresis (an electric field) is very simple. A controlled electric field is applied via electrodes immersed in the sample suspension and this causes the charged particles to move towards the electrode of opposite polarity. Viscous forces acting upon the moving particle tend to oppose this motion and an equilibrium is rapidly established between the effects of the electrostatic attraction and the viscous drag. The particles therefore reach a constant "terminal" velocity. This velocity is dependent upon the electric field strength or voltage gradient, the dielectric constant and viscosity of the liquid - all of which are known - and the zeta potential. It is usually expressed as the particle mobility which is the velocity divided by the unit field strength.



Graph of the isoelectric point.

Isoelectric Point

Is the pH at which a colloid carries no net charge. Below the isoelectric point particles carry a net positive charge, above it a net negative charge.

Due to a preponderance of weakly acid residues in almost all proteins, they are nearly all negatively charged at neutral pH. The isoelectric point is of significance in protein purification because it is the pH at which solubility is often minimal and at which mobility in an electrofocusing system is zero (and therefore the point at which the protein will accumulate).

Colloidal Stability

Colloidal suspensions are stabilized in one of two ways. Surface charge, naturally occurring or added, enhances electrostatic stability. Adsorption of nonpolar surfactants or polymers enhances stability through steric stabilization. Electrostatic stabilization gives rise to a mobile, charged, colloidal particle whose electrophoretic mobility can be measured. Zeta potential is calculated from mobility. The square of the zeta potential is proportional to the force of electrostatic repulsion between charged particles. Zeta potentials are, therefore, measures of colloidal stability. Increasing the absolute zeta potentials increases electrostatic stabilization. As the zeta potential approaches zero, electrostatic repulsion becomes small compared to the ever-present attractive Van der Waals forces. Eventually, instability increases, which can result in aggregation followed by sedimentation and phase separation.

Services

- Single Point
- Titration
- Iso-electric point determination
- Aqueous and non-aqueous media
- High Ionic Strength/Viscosity

Molecular Weight Analysis

Molecular weight and size are two key properties of all macromolecules including biologically important and synthetic polymers. Performance and processability are directly related to these parameters. Light scattering is capable of absolute measurements in either batch or flow mode. For distribution information, a light scattering detector is couple to a GPC/SEC instrument to a light scattering detector.

Macromolecular samples often have a range of molecular weights. In some cases, this distribution is quite narrow, and in other cases, it is very broad and/or multimodal. Variations in the distribution can indicate the presence of impurities or aggregation. However, for other applications, the average molecular weight is sufficient to characterize your sample. Sometimes the distribution is of interest, and sometimes the overall average is sufficient.

Static Light Scattering, GPC Method

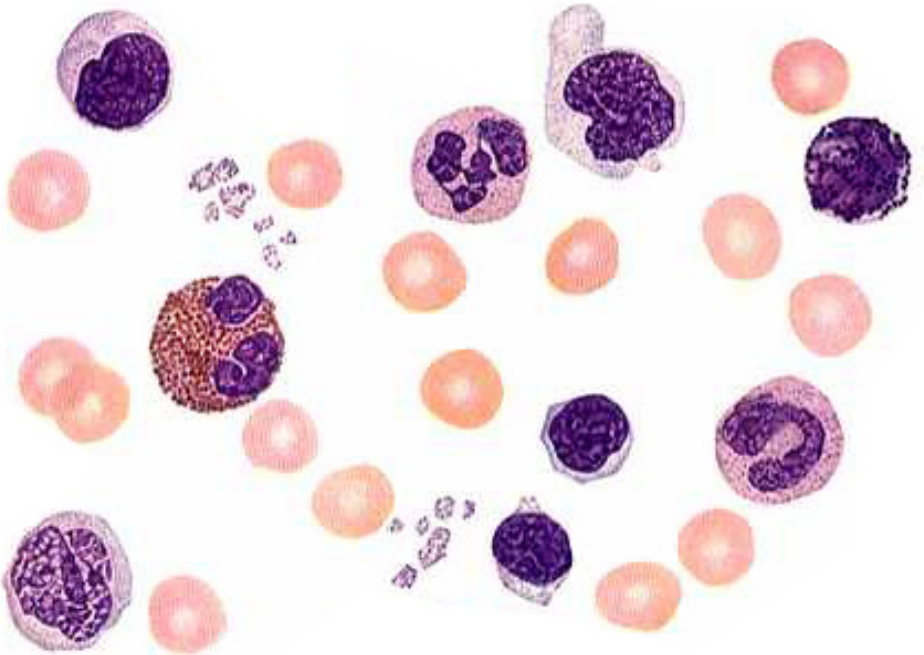
Size exclusion chromatography (SEC), also known as gel permeation chromatography (GPC), uses columns to separate polymer samples. In the standard method columns are calibrated to obtain a relationship between molecular weight and elution volume. However, the column-sample interaction depends on not just the size (molecular weight) but also the chemistry of the sample. Therefore, an accurate column calibration requires standards over a range of molecular weights with exactly the same chemistry and structure (e.g., branching) as the sample. With a few exceptions, such standards are difficult or impossible to obtain, especially for new or unique materials.

Utilizing a light scattering detector in line with the GPC system provides a method of determining absolute molecular weights without resorting to any assumptions about the sample or column calibration. In this configuration the column is then simply used to separate the species of interest and the light scattering detector gives full information on the molecular weight of each fraction.

Static Light Scattering, Batch Method

The average molecular weight of a sample may be characterized utilizing batch mode techniques to easily determine the samples properties. Batch mode analyses permit determination of the intensity average molecular weight, M_w , the z-average radius of gyration, R_g , and the second virial coefficient, A_2 . Data may be presented in the Zimm, Berry, or Debye plots formats to calculate these parameters.

Molecular weights in batch mode analyses are conducted by preparing dilute sample solutions of known concentrations.



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