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APPARATUS AND PROCESS FOR DETERMINING PARTICLE SIZE BY X-RAY ABSORPTION ANALYSIS

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ABSTRACT OF THE DISCLOSURE

An apparatus and process for measuring and automatically recording the particle size distribution of material in suspension, which is based on the application of Stokes' Law of sedimentation. A beam of X-rays is used to measure particle concentration in terms of the transmittance of the beam in the suspension relative to the suspending fluid. A programmed drive mechanism causes the distance between the point of intersection of the X-ray beam with the suspension and upper or lower surface of the suspension to be continuously changed with time.

Background of the invention

The present invention relates to a particle size analyzer. It also concerns a unique process for automatically measuring the particle size distribution of a dispersion of finely divided material.

The processes for fine particle size measurement that are in the literature can be broken down into two main headings: (1) methods yielding average particle size only, which is of limited interest, since a particulate material can rarely be fully characterized in terms of its average particle size; and (2) methods yielding size distribution.

Methods yielding particle size distribution have been extensively treated in the literature. They are represented by the following five techniques: sieving; direct optical or electron microscopic counting and measuring; electronic counting and sizing; elutriation; and sedimentation. Most of these techniques have limited use. Thus the sieving technique is not readily applicable for measurement of particles finer than 50 microns; it requires a sizeable sample, much time and many manual operations; it also gives questionable results for anisometric particles. A serious disadvantage of the optical technique lies in the fact that the transmission of light through a suspension of fine particles is dependent on the size and color of the particles as well as their concentration; it further requires a lengthy mathematical reduction of the primary data.

The direction microscopic technique is tedious because of the statistical requirements for counting and measuring huge numbers of particles; it further requires lengthy mathematical analysis of the results. The electronic technique is complex and expensive and also difficult to employ with fine particle material having a wide particle size distribution; it also requires a rather lengthy and tedious mathematical reduction of the primary data to obtain the final particle size distribution. Particle size analysis by elutriation is very difficult to control experimentally.

Today, the sedimentation rate technique forms the basis of most of the practical methods of particle size

measurements. This technique is dependent on the fact that the rate of fall of a particle through a viscous medium is directly related to the size of the particle by Stokes' Law. Stokes' Law for spherical particles is expressed as

$$D=Kv^{1/2} \qquad (1)$$

where D is the diameter of a falling sphere and v is its equilibrium rate of fall (velocity). K is a constant depending on the density ρ (rho) of the sphere, and on the density ρ_o (rho$_o$) and viscosity η (eta) of the viscous medium, K being equal to

$$K=\left(\frac{18\eta}{(\rho-\rho_o)g}\right)^{1/2} \qquad (2)$$

where g is the gravitational acceleration.

In practice, truly spherical particles are uncommon, and it is recognized that Stokes' Law is not exact for any other shape; however, the deviations are small in most cases. Since irregular shapes cannot in any case be described by a single linear dimension, it is an accepted practice to specify the size of irregular or nonspherical particles in terms of the diameter of a sphere of the same material that would have the same sedimentation velocity. This diameter is commonly referred to as "Stokes' diameter" or "Equivalent Spherical Diameter."

In general in sedimentation analysis a dilute (less than 10% solids) deflocculated suspension of a fine particle material is stirred to render it homogeneous. It is then allowed to stand tranquilly while undergoing sedimentation. Time is measured from the beginning of the settling period. By Stokes' Law, a particle of diameter D will settle a distance h in time t according to the expression

$$D=K\left(\frac{h}{t}\right)^{1/2} \qquad (3)$$

where K is as in Equation 2 supra. Consequently, after a given time t_i all particles larger than the corresponding value D_i will have fallen below a distance h from the surface of the suspension. If the initial concentration of particles is C_o g./ml. and the concentration after time t_i at distance h is C_i g./ml., then the percent of particles P_i, by weight of sample finer than D_i is

$$P_i=100\frac{C_i}{C_o} \qquad (4)$$

Various methods for determining particle size distribution by sedimentation employ the above relationships, but differ in the specific technique used to determine C_i as a function of time at a known h. Thus in the Andreasen Pipette method, a small sample of suspension is periodically withdrawn by means of a pipette from depth h, and the concentration of solid determined. In certain ASTM and TAPPI methods, e.g., TAPPI, T649 s.m. 54 and ASTM D422–61T, a hydrometer is periodically inserted into the suspension and the concentration remaining at the bulb depth, h, is determined from the specific gravity registered.

The above procedures suffer from several serious faults. For example, these procedures do not measure concentration at a specific value of h, but rather over a considerable range around some mean value of h. This has an averaging effect that "smears out" any detail in any distribution curve. Furthermore, repeated removal of samples, or insertion of the hydrometer, during a size