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A Phtometric Particle-size Analyzer with a Moving Light Flux

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A recording, photometric, particle-size analyzer has been developed which is useful in measuring the particle-size distributions of powdered materials in the subsieve range. Provision for continuously moving the optical system upwards allows for rapid analysis of the finer particles, while by moving the system downwards accurate analysis of the particles with a larger density is possible. It was ascertained that the movement of the optical system has no effect on the results obtained.

The photo-extinction method of size-frequeny determination offers great possibilities, as has been pointed out by Rose,[1] for the development of a precise and convenient technique applicable to both routine and research investigations. As a matter of fact, however, this method takes a long time for a routine application, just as do the other sedimentation methods of size-frequency determination. Talcite and Paulus[2] constructed a recording, photometric, particle-size analyzer which has a provision for continuously varying the sedimentation depth so that the analysis can be carried out within an hour. However, they did not clarify how the movement of the sedimentation vessel affects the results obtained, and their system may be accompanied by trouble resulting from the movement of the vessel. We have now constructed an analyzer provided with an apparatus for continuously moving the light flux upwards or downwards, in order thus to carry out a rapid analysis of the finer particles or an accurate analysis of the particles with a larger density. The optical system designed to move smoothly allows a complete, accurate analysis to be made within an hour. The comparison of the results obtained by means of an apparatus with a moving light flux with those obtained by the ordinary static method makes it clear that the movement of the optical system has no effect on the analysis.

Principles.—The principles of the photo-extinction method have been published elsewhere.[1] The absorbance of a powder suspension is related to its concentration by an extension of the Lambert-Beer law:

$$\ln (I_0/I) = kcl \sum_{d_0}^{d_1} K_x n d_x^2 \qquad (1)$$

where I_0=the intensity of the incident light; I= the intensity of the transmitted light; k=a factor equal to A_p/d_x^2, where A_p is the projected area of a particle, and d_x, the diameter; c=the concentration of the powder suspension (g./ml.); l=the length of the cell; K_x=the extinction coefficient, that is, the ratio of the light-obscuring power of a particle to that for the same particle if the "square law" of geometrical optics were applicable, and n= the number of particles, whose diameter is d_x, per gram of powder. When we begin with a uniform suspension and a transverse light beam at a fixed depth below the surface, no change in the absorbance of the suspension will occur until the largest particles in the suspension fall from the surface to the level of the measuring beam. At this time the absorbance will decrease by an amount proportional to the concentration of the largest particles. Any point on the absorbance-vs.-time curve represents, then the concentration of particles smaller than the particle which would fall the distance, h, from the surface to the measuring beam during the time, t. When the light flux moves upwards or downwards, two cases may be considered:

a) when the light flux moves upwards with a constant velocity, v_0, from a point whose depth is b below the surface of the suspension, then:

$$h = b - v_0 t$$

and: $$d_x = K\sqrt{v} = K\sqrt{h/t};$$

Then: $$t = K^2 b/(d_x^2 + K^2 v_0);$$

b) when the flux moves downwards with a constant velocity, v_1, from a point whose depth is b below the surface of the suspension and when, after a time, t_1, it moves upwards with the same velocity, then:

$$h = v_1 t + b \qquad \text{for} \quad t < t_1$$

$$t = K^2 b/(d_x^2 - K^2 v_1)$$

or: $$h = v_1(2t_1 - t) + b \qquad \text{for} \quad t > t_1$$

$$t = K^2(2v_1 t_1 + b)/(d_x^2 + K^2 v_1)$$

where: $K^2 = 18\eta/g(\rho_p - \rho)$,

and η=the viscosity of the medium (poise), ρ_p= the density of the particle (g./ml.), ρ=the density

1) H. E. Rose, "Measurement of Particle Size in Very Fine Powders," Constable, London (1954).
2) N. A. Talvite and H. J. Paulus, *Rev. Sci. Instr.*, **27**, 763 (1956).

of the medium (g./ml.), and g=the gravitational constant (cm./sec./sec.).

A size-distribution based on the weight is expressed in the form of:

$$F_x = \frac{d_m}{K_m}(\ln I_1 - \ln I_2) \Big/ \sum_0^\infty \frac{d_m}{K_m}(\ln I_1 - \ln I_2),$$

where I_1 or I_2 is the intensity of the transmitted light for the suspension containing the powder whose diameters are in the range from 0 to d_1 or d_2 respectively. d_m is the mean of d_1 and d_2, and the K_m is the K_x value for d_m. I_1 and I_2 are proportional to the photocurrents, M_1 and M_2, from the photomultiplier.

Thus a size-distribution curve is obtained with an absorbance coordinate representing the concentration of particles less than the stated size and a time ordinate representing the particle size in terms of the Stokes diameter.

Design Constructions.—The analyzer is arranged as shown in the schematic diagram of Fig.

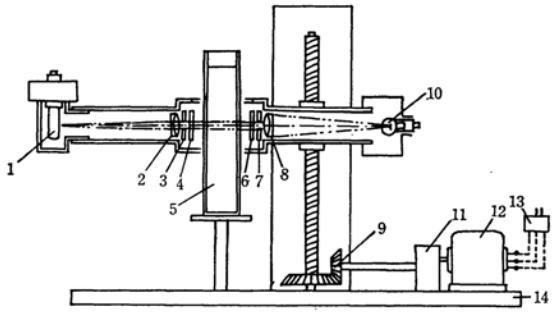

Fig. 1. Schematic diagram of the photometric particle-size analyzer.
1: Photomultiplier
2: Telescope lens
3: Slits
4: Absorption filter
5: Sedimentation cell
6: Monochrome filter
7: Slit
8: Collimeter lens
9: Gear for movement of light flux
10: Light source
11: Gear box
12: Synchronous motor
13: Reversing Switch
14: Base

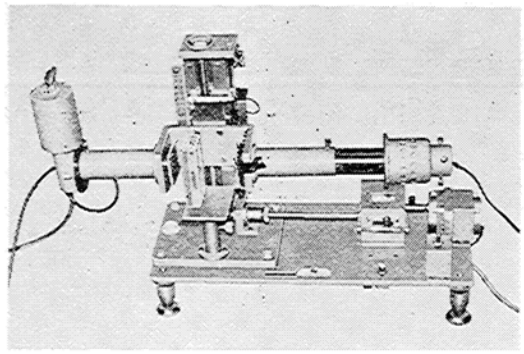

Fig. 2. Picture of the analyzer.

1. The apparatus is pictured in Fig. 2. The light source is a 5-amp., 6-V. bulb with a straight coiled filament and operated at a rated voltage from a 6-V. storage battery, the current being controlled with a rheostat. The collimation of a light beam is required for accurate analysis; therefore, the beam is made parallel by a condenser lens at its focal distance from the light source, while a slit immediately before the sedimentation cell limits the dimensions and angular divergence of the beam. A second slit following the cell cuts off the scattered light. A filter mount is located between the cell and the second slit, and a neutral filter is used for the control of the light intensity, while an interference filter is mounted when monochromatic light is required. The distance from the light source to the sedimentation cell is 33 cm., and that from the cell to the surface of the photomultiplier is 22 cm. The sedimentation cell must have parallel sides; its usual size is $40 \times 15 \times 100$ mm. The applied voltage for the photomultiplier, RCA-931A, is 700 V. to 770 V. The photocurrent is recorded by a Honeywell recorder.

The slit width is fixed at 25 mm., while its height may be varied in two fixed steps; 1 mm. and 2 mm. The optical system, including the light source, the lens, the slits and the photomultiplier, is mounted on a steel base arm and is fastened to a vertical brass slide. It can be raised or lowered by means of a standard micrometer head; this micrometer is coupled, by way of a set-screw clutch and right-angle drive, to a synchronous motor geared to raise or lower the

TABLE I. EXPERIMENTAL CONDITIONS

Exp. No.	Sample	Density of powder in the suspension wt.%	Conditions for movement of light flux					
			Movement	Velocity cm./sec.	Position of start cm.	Position of reverse cm.	Position of stop cm.	Time for measurement, min.
305	Silica powder	0.5	Fixed	0	4	—	4	30
306	Silica powder	0.5	Down- and upwards	0.0111	2	4	0	9
309	Silica powder	0.5	upwards	0.0333	10	no reverse	0	5
311	Silica powder	0.5	upwards	0.0111	6	no reverse	0	9

Table II. Calculating process of result obtained

d_x, μ	0	5	10	15	20	25	30	35	40	45	50	
K_m		2.70	2.10	1.95	1.80	1.65	1.50	1.45	1.40	1.35	1.3	1.2
t, sec.		725.8	466.3	292.2	191.9	113.2	73.78	52.28	39.12	30.44	24.39	
M_x	8.19	7.50	6.87	4.32	3.70	3.42	3.25	3.17	3.12	3.095	3.08	3.04
$\log M_x$	0.9133	0.8751	0.8370	0.6355	0.5682	0.5340	0.5119	0.5011	0.4942	0.4907	0.4886	0.4829
$\log \frac{M_{x-1}}{M_x}$		0.0382	0.0381	0.2015	0.0673	0.0342	0.0221	0.0108	0.0069	0.0035	0.0021	0.0057
$\frac{1}{K_m}\bar{d}_x \log \frac{M_{x-1}}{M_x}$		0.035	0.136	1.292	0.654	0.466	0.405	0.242	0.185	0.110	0.077	0.285
$\sum_{d_x}^{\infty}\left(\frac{1}{K_m}\bar{d}_x \log \frac{M_{x-1}}{M_x}\right)$		3.887	3.852	3.716	2.424	1.770	1.304	0.899	0.657	0.472	0.362	0.285
$\sum/\sum t \times 100$		99.1	95.6	62.4	45.5	33.5	23.1	16.9	12.1	9.3	7.3	
F_x		0.9	3.5	33.2	16.9	12.0	10.4	6.2	4.8	2.8	2.0	7.3

Temp.: 25.0°C

optical system. The rate may be varied in three fixed steps; 0.033 cm./sec., 0.011 cm./sec., and 0.0037 cm./sec. A reverse microswitch automatically changes the movement of the optical system from downwards to upwards when the system reaches the surface of the fixed height in the sedimentation cell, while a second microswitch shuts off the motor at the end of the run and may also shut off the paper drive of the recorder.

A 50 cm. × 26 cm. × 1 cm. structural, channel-steel plate forms the base of the analyzer; a rigid mounting surface for the sedimentation cell is fixed upon this base.

Operation.—Silica sand (density: 2.66) and tungsten carbide powder (density: 14.0) were used as the two samples for the test of the analyzer. The mixing of the suspension, whose concentration of solids was varied in several steps, was accompanied by quick inversions and agitations of the cell and by returning it to the cell component without delay. Sodium hexametaphosphate (0.2% of the total volume) was added to the suspension prior to the mixing in order to obtain a good dispersion. The intensity of the light beam was adjusted and the recorder chart and the mechanism which moves the optical system were actuated beforehand in order for the record to begin to operate at the instant when the cell was returned to its compartment. The chart speed was 0.0423 cm./sec. When the optical system is lowered in the first period of the experiment, the reverse switch changes the movement at the time of the arrival of the system at the fixed surface, its height from the surface of the suspension being measured before the start of the experiments. On the arrival of the light flux at the suspension surface, the measurement is finished.

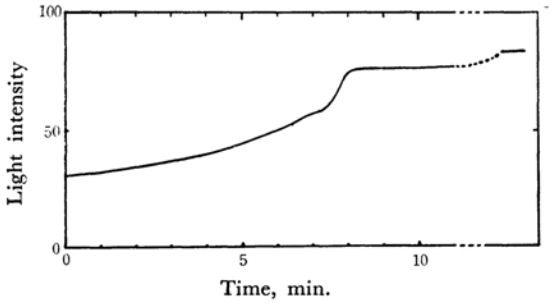

Fig. 3. Example of the chart obtained.

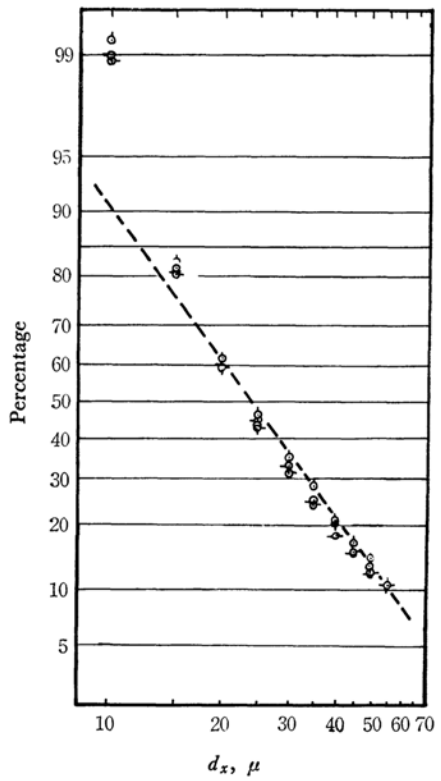

Fig. 4. Typical curves of particle-size distribution.

⊙ : A Fixed light flux.
⊙–: B Moved upwards b=6 cm.
⊙ : C Down- and upwards
–⊙ : D Moved upwards b=10 cm.

Performance.—The experimental data obtained under the conditions shown in Table I were calibrated in terms of Stokes' diameter. A typical procedure is shown in Table II, which relates the description to the principles. The values of K_m, the extinction coefficient, are those published by Rose, while M_x is the reading of the photocurrent for the suspension of the powder whose diameter is d_x. The summation of the values of $\{1/K_m \cdot d_x \cdot \log(M_{x-1}/M_x)\}$ was carried out from right to left in the table, that is, beginning with the value for the largest particles. The chart obtained in the experiments is shown in Fig. 3. Typical curves of particle-size distribution are shown in Fig. 4. In this figure it may be seen that the movement of the light flux has no effect upon the particle-size distribution curves. The deviation of the measured values with the movement of the light flux against those in the case of a fixed flux was very small and was estimated to be within the range of experimental error. It might be due to the disturbance of the suspension which remains immediately after the mixing of the powder suspension; this may cause the portion with the smaller diameter to increase. Such a deviation may occur when the analysis is carried out without taking the precaution of suppressing the disturbance of the suspension. Experiments are now being carried out in our laboratory in order to clarify this phenomenon. The fluctuations rise from other sources than the movement of the light flux, such as the inhomogeneity of the dispersion and the fluctuation of the cell temperature. The result for tungsten carbide powder seems to be more accurate in the subsieve range when the light flux is moved downwards at the beginning of the measurement than in the case of a fixed flux. In this case it was possible to shorten the measuring time to 9 min. by moving the flux, while the measurement with a fixed flux takes 30 min. All the measurements shown in Table I were carried out in 10 min., and the calculation for one measurement was about 10 min. longer than in the case of a fixed flux. The latter would be shortened by the use of an appropriate calculating chart.