

# PARTICLE-SIZE STUDIES

## PROPERTIES OF FINELY DIVIDED MATERIALS

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A survey of the literature on particle-size studies indicated the desirability of classifying particle properties according to their influence on other properties. Such a classification was made, and experimental data were discussed on the basis of this classification. A study of five closely fractionated materials, having different particle shapes and about 50 microns in size, was made in which it was shown that the two mean microscopic diameters based on the square root of the projected area and the shorter of the two visible dimensions at right angles to each other gave results essentially the same for silica, coal, slate, and galena, while for mica the latter microscopic diameter gave results equal to 0.9 of the former. The mean microscopic diameter was found to vary from 1.2 to 1.5 times the sieve aperture and from 1.1 to 2 times the equivalent sedimentation diameter, depending upon the material. The mean sedimentation diameter of materials just passing a sieve may be from 0.8 to 1.2 times the sieve aperture, but the point of separation of a No. 325 sieve for combined sieving and sedimentation analyses may be taken as 53 microns for many materials. It was pointed out that the computation of specific surface from size-distribution data, using shape constant ratios, rather than from surface and volume constants would simplify the problem considerably.

Particle-size studies in the range of sizes from the finer sieve sizes of about 75 microns down to about 0.2 micron are of increasing industrial value because the variations in particle properties affect the production and use of such materials. (The colloidal range which is predominantly finer than 0.2 micron, although approaching this size as an upper limit, is not included in the scope of this work.) Although there have been many publications on certain specific properties of particles (such as size or shape), the results have never been assembled and studied in relation to other particle properties. The object of this paper was to review the more important studies and to classify particle properties according to their influence on other properties. Certain experimental data were then studied on the basis of this classification.

A single particle may be evaluated in terms of three primary functions: dimension (or size), dimensional relationships (or shape), and surface configuration (or structure). If a material consists of more than one particle and a range of sizes, the additional primary property of size distribution must be considered. These primary properties control the secondary properties, such as specific surface, the packing characteristics, flocculation in suspensions, obscuring power to radiations, and chemical activity. In the following classification certain of the above properties have been discussed on the basis of experimental results for different types of materials.

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## I. PRIMARY PROPERTIES

## A. PARTICLE SIZE

1. *Microscopic diameters*

Observation of finely ground particles under the microscope shows the necessity of defining particle size by some arbitrary diametric dimension, since very few materials are spherical in shape. Among the most important diameters that are used to define the size of particles of irregular shape are the following: (a) the diameter of Green (23, 24), which is a statistical diameter,  $d_g$ , taken in one direction across the particles lying on the same line across the field; (b) the diameter of Heywood (26, 27, 28), which is the diameter,  $d_h$ , of a circle having the same projected area as the particle; (c) the diameter of Wadell (48), who uses a true nominal diameter,  $d_n$ , which is the diameter of a sphere having the same volume as that of the particle; (d) the diameter of Martin (34), which is similar to that of Green except that the diameter,  $d_m$ , is that which divides the projected area of the particle into two equal areas; (e) the diameter of Perrott and Kinney (36), in which the diameter,  $d_k$ , is the side of a square having the same area as the projected area of the particle; (f) the diameter of Weigel (52), which is the visual average,  $d_w$ , of the two visible dimensions; (g) the diameter of Work (55), who used a diameter,  $d_b$ , which is the "intermediate" dimension, being the shorter of the two visible dimensions at right angles to each other; (h) the diameter of the preceding author, who suggested a mean spherical diameter,  $d_v$ , which is the diameter of a sphere having the average weight of the particles; (i) the diameter of Andreasen (2), which is the cube root,  $d_a$ , of the volume of the particle. Other diameters that have been used are: (j) a harmonic mean diameter,  $d_s$ , which is equal to three times the reciprocal of the sum of the reciprocals of the three individual dimensions (length, width, and thickness); (k) a diameter,  $d_c$ , equal to the cube root of the product of the three dimensions; and (l) a diameter,  $d_r$ , equal to the arithmetic average of the three dimensions at right angles to each other.

Perrott and Kinney (36), using coal, have shown a comparison of various types of microscopic diameters computed to weight distribution. These authors have also compared results obtained using their diameter ( $d_k$ ) with those obtained by water sedimentation and air elutriation; for the 0-7.5 micron fraction the last two methods agree, but the microscope gives low results. They also showed that, for coal,  $d_c$  is essentially equal to  $d_r$  and both do not vary greatly from  $d_k$ . They showed further that the weight calculated from  $nd^3$  is about 1.5 times the actual weight but varies from 1.4 to 1.8 times as much. This would be expected, since the volume of a cube is  $(6/\pi) = 1.92$  times as great as that of a sphere the diameter of which is equal to one edge of a cube; this is also the ratio of surface. The ratio becomes less as the particle approaches a sphere in shape and all three dimensions are averaged.

Weigel (52) has used  $d_w$  for studies on various types of mineral fillers and has given a comparison of sizes by count with results on settling velocity. This author computed the mean diameter of the material from  $\Sigma nd^3 / \Sigma nd^2$  (where  $n$

is the number of particles of diameter  $d$ ) and for talcs, micas, and slates applied a percentage correction to the diameter. Heywood (28) has shown that  $d_a/d_h$  may vary from 1.3 for rounded sand to 7.0 for mica in coarse sizes. A comparison of  $d_k$  and  $d_h$  shows that the calculated ratio of  $d_h/d_k$  is equal to

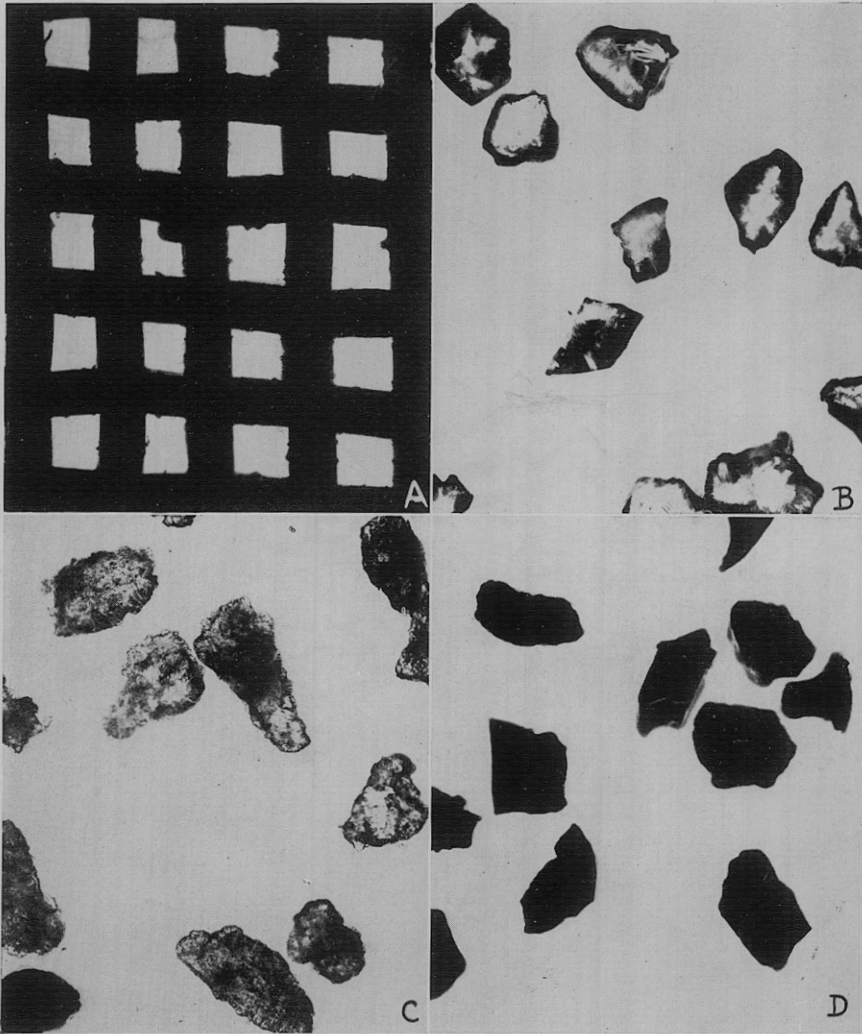


FIG. 1. Photomicrographs: A, No. 325 sieve; B, silica; C, slate; D, coal; E, galena; F, mica; G, scale, one division equals 10 microns; H, diatomaceous earth.

$(2/\sqrt{\pi}) = 1.13$ . Patterson and Cawood (35) have described a graticule for oculars containing etched circles which might be useful in measuring  $d_h$ . Work (55) has shown  $d_v$  to be from 0.9 to 1.0 times the sieve aperture for large sizes. Bennett (7) has shown an equivalent "projected diameter" to be 1.41 times the sieve aperture of square sieves and 1.21 times the aperture of round holes, "as

suggested by Heywood." Roller has stated that the ratio of  $d_r/d_e$  is generally less than 1.06 and that the arithmetic mean may therefore be used in place of  $d_e$ . Gadd (20) has stated that  $d_m$  is about the same as  $d_r$ . Weinig (53) has proposed a method of computing surface based on a comparison of the particle with a

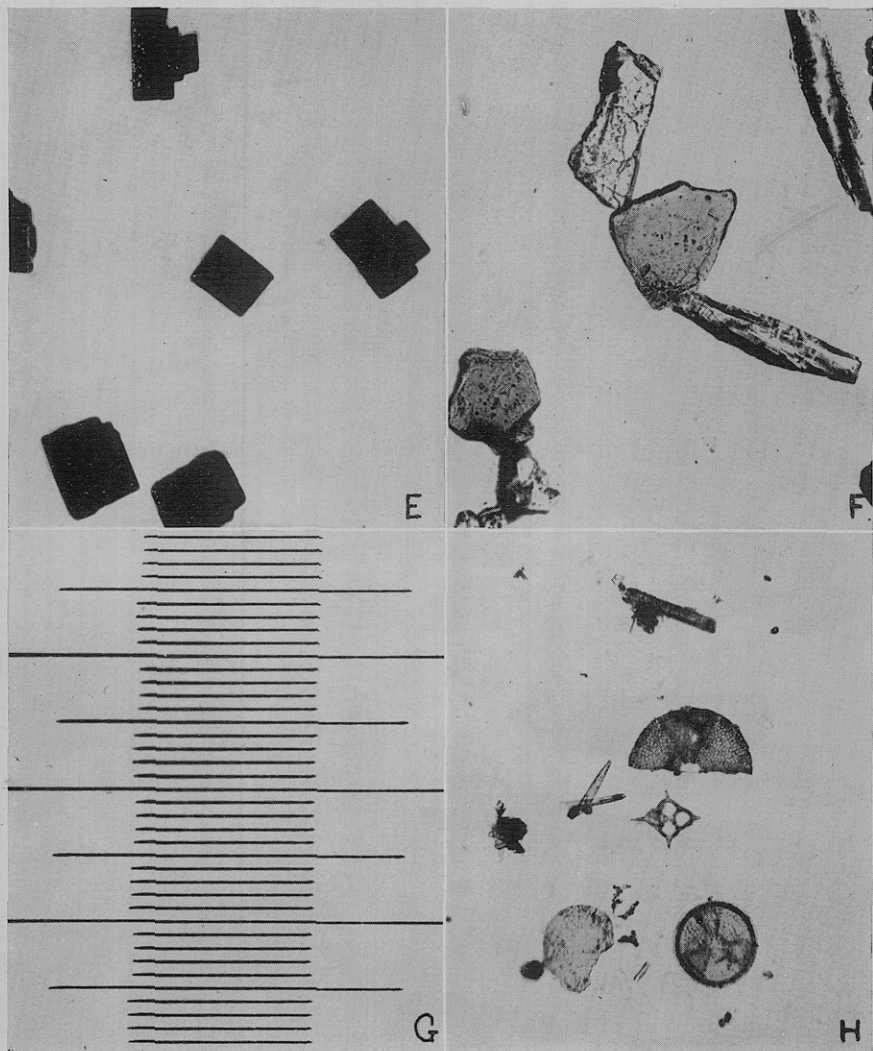


FIG. 1—concluded

cubical configuration. These shape constants approach a constant value in the No. 65 to No. 100 sieve range and are similar for different types of crushing operations.

The preceding review of the literature indicates the numerous ways in which microscopic dimensions may be reported. Results of different investigators

cannot be compared unless the relations among the various diameters are known, and few are. Microscopic analysis is, at best, a laborious and time-consuming operation, and it seems pertinent to suggest that standard procedures be used. The most simple microscopic diameters to determine are those of Perrott and Kinney ( $d_k$ ) and of Work ( $d_b$ ). Experience with the former has indicated its general adaptability, but the latter is somewhat more rapid to use. For this reason the following experiments were carried out to ascertain the relations between the two. Five materials representing particle shapes frequently encountered in practice were selected and subjected to the following operations to obtain uniform fractions. Fifty to 100 g. of finely ground powder were weighed and sieved through a No. 325 sieve, using a water-spray nozzle (4) at 10 pounds per square inch pressure for 10 min. The residues were dried and weighed and

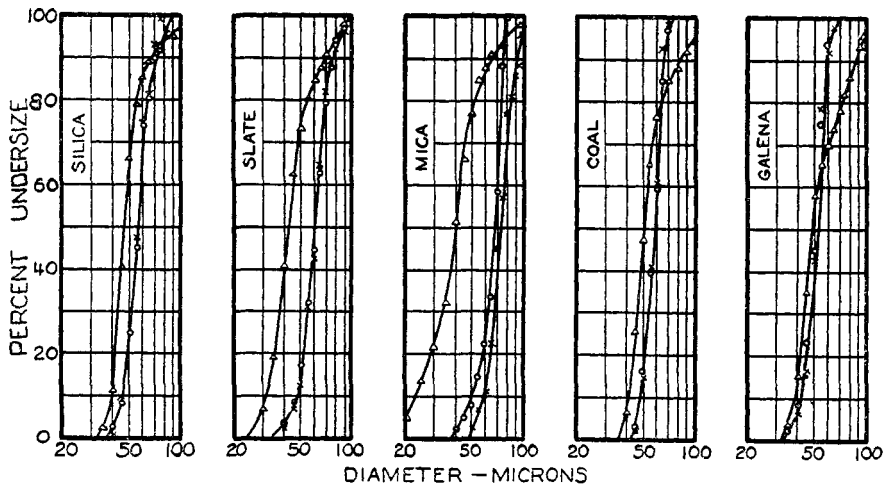


FIG. 2. Size distribution curves:  $\times$ ,  $d_k$ ;  $\circ$ ,  $d_b$ ;  $\Delta$ , turbidity

then placed on a new No. 325 sieve and sieved dry for 5 min., using a mechanical shaker of the vibratory type. The material passing was recovered and used for study. It consisted of a closely fractionated product that would pass a No. 325 sieve but not when sieved wet.

The above procedure was used for the following different materials: a ball-mill-ground Ottawa sand, D-2; a ring-roller-mill-ground green slate, 470; a green mica, 432, method of processing not known; and ball-mill-ground samples of coal, C, and galena, G. These five materials cover particle shapes representative of the acicular (slate), angular (coal), platelike (mica), regular cubical (galena), and angular to rhombohedral (silica) types of particles. The characteristic shapes of these materials are illustrated in figure 1. Figure 1A is a photomicrograph of a portion of the sieve used for dry sieving. Figure 1H is a photomicrograph of diatomaceous earth retained on a No. 325 sieve, using the wet sieving procedure.

TABLE 1  
Comparison of data on 325-mesh material and certain other materials

MATERIAL	NO.	$D_s$ IN GRAMS PER CUBIC CENTI- METER	PER CENT PASSING A No. 325 SIEVE		325-MESH MATERIAL	MEAN DIAMETER*					SPECIFIC SURFACE† IN SQUARE CENTIMETERS PER GRAM			
			(a)	(b)		(c)	$d_k$ (e)	$d_b$ (e)	$d_s$ (f)	$d_t$ (g)	$d_h$	$S_k$	$S_b$	$S_s$
Silica.....	D-2	2.67	78.8	77.8	1.87	57	58	48	48	58	66K	65K	78K	79K
Green slate....	470	2.82	96.4	93.8	2.50‡	62	62	38	42	58	57K	57K	93K	85K
Green mica....	432	2.92	82.5	80.7	0.52	73	66	35	35	27	47K	52K	99K	99K
Coal.....	C	1.75	73.4	64.1	4.84	59	59	50	52	50	97K	97K	114K	111K
Galena.....	G	7.66	65.3	57.6	7.26‡	52	52	48	50		27K	27K	29K	26K
Red slate.....	642	2.80	96.4							47				
Silica.....	196	2.62	94.8							50				
Silica.....	W-1	2.67	29.7				62§			51				
Silica.....	F-14	2.67	98.0				58§			58				
Traprock.....	113	2.91	83.4							51				

\* Diameters:

$d_k$  = diameter based on square root of projected area,

$d_b$  = diameter based on shorter of two visible dimensions at right angles to each other,

$d_s$  = diameter based on sedimentation,

$d_t$  = diameter based on turbidity data, and

$d_h$  = diameter read from sedimentation curves of whole powder at per cent undersize found under (a).

† The subscripts under specific surface refer to the corresponding surface mean diameter.  $K$  is the shape constant ratio which may vary for different materials.

Column headings:

(a) Sieve test: 1-g. sample on 2-in. sieve with 10 lb. per square inch nozzle pressure for 1 min.

(b) Sieve test: 50-g. sample on 8-in. sieve with 10 lb. per square inch nozzle pressure for 10 min.

(c) Per cent passing a No. 325 sieve dry but retained wet; 50-g. sample.

(e) Calculated from  $\Sigma nd^3/\Sigma nd^2$ .

(f) From midpoint of sedimentation curve in region of greatest slope.

(g) Calculated from turbidity specific surface.

(i) From turbidity data, obtained by dividing the specific surface obtained by 6; the latter is the shape constant ratio which is assumed in this method.

‡ 100-g. sample.

§ Read from microscopic count of whole powder at per cent undersize found under (a).

TABLE 2  
Comparison of various particle diameters for different materials

MATERIAL	$d_b/44$	$d_s/d_b$	$d_t/44$
Silica, D-2.....	1.32	0.83	1.09
Slate, 470.....	1.41	0.62	0.87
Mica, 432.....	1.50	0.53	0.80
Coal, C.....	1.34	0.85	1.09
Galena, G.....	1.18	0.92	1.14

A microscopic count at 5-micron intervals was made, using five fields from each of two slides to give a minimum of one hundred particles counted. From these data a weight distribution for the Work diameter,  $d_b$ , and the Perrott and Kinney diameter,  $d_k$ , could be computed as well as the surface mean diameter. The weight distribution was calculated from  $nd^3/\Sigma nd^3$  and the surface mean diameter from  $\Sigma nd^3/\Sigma nd^2$ , where  $n$  was the number of particles of diameter  $d$ . The weight distributions are shown in figure 2 and the mean diameters are given in table 1.

Study of the data shows, with the exception of the mica, that the diameter of Work,  $d_b$ , and that of Perrott and Kinney,  $d_k$ , give essentially the same values both for distribution and for surface mean diameter. In the case of the mica the ratio of  $d_b/d_k$  is about 0.9.

The data for these materials also show (table 2) that the  $d_b$  diameter of the particles just passing a No. 325 sieve (nominal opening of 44 microns) may be from 1.18 to 1.50 times as great as the aperture.

## 2. Sedimentation diameter

With the exception of sieve analysis and microscopic methods, practically all of the methods used for determining particle-size distribution define the size in terms of the sedimentation diameter. This is the diameter of a sphere that settles at the same velocity as the particle being studied.

In general, isothermal free settling of particles may be assumed to follow this form of Stokes' Law:

$$d = \sqrt{\frac{18\eta v \times 10^8}{(D_s - D_l)g}} \quad (1)$$

where  $\eta$  = viscosity, in poises,  
 $v$  = terminal velocity,  $h/t$ ,  
 $h$  = distance of fall, in centimeters,  
 $t$  = time of fall, in seconds,  
 $g$  = gravitational constant in centimeters per second per second,  
 $D_s$  = density of particle (greater than  $D_l$ ), in grams per cubic centimeter,  
 $D_l$  = density of liquid, in grams per cubic centimeter, and  
 $d$  = diameter of a sphere in microns.

For irregular shaped particles,  $d$  is the diameter of a sphere that falls at the same rate as the particle. This equation is valid for Reynolds numbers below 1 where the Reynolds number is defined (51) as:

$$Re = \frac{vdD_s}{\eta \times 10,000} \quad (2)$$

While numerous studies on Stokes' Law have been published, there are few on the application of it to the practical problem of relating settling velocity and the diameter of subsieve particles of irregular shape. These relations are of importance where the conversion of particle-size data is required, as, for example, in the computation of the surface area of a powder from its weight distribution.

The analysis of the settling of particles generally takes the form of a plot of a modified Reynolds number against a coefficient of resistance with parameters for variation in shape. Wadell (49) has developed such curves for the "true nominal diameter," which is the diameter of a sphere having the same volume as that of the particle. In another article by this author (50) it is indicated that equivalent diameter (Stokes' Law) and true nominal diameter are nearly the same at low Reynolds numbers. Methods are given for computing equivalent diameters and equivalent velocities by combining equations to eliminate diameter or velocity, as the case may be.

Recent papers on the validity of Stokes' Law are those of Dorr and Roberts (15), who showed its limit to be at about Reynolds numbers of 1 and gave a modified equation for higher Reynolds numbers, and that of Squires and Squires (41), who showed that the settling of thin discs may vary, depending upon the orientation of the particle relative to the direction of motion. They stated, further, that discs and spheres settle at the same velocity only when the ratio of the diameter of the disc to its thickness is 1.77 for discs settling with face perpendicular to the direction of motion; in this respect they disagree with Wadell (50). Svedberg (43) has published a table showing calculated relative velocities for disc- and rod-shaped ellipsoid particles for various axes ratios; the values showed a variation of from about 0.75 to 1.0 times the velocity of fall for spheres. He also mentioned the wall effect for settling, with an indicated correction of 1 per cent for particles 0.01 of the distance between the enclosing walls. The wall effect has also been discussed by Squires and Squires (41) and by Wadell (50). Marshall (33) has discussed orientation and shape effects in settling and has stated that shape can be neglected for length-to-thickness ratios of 5 to 1 only if the volume of the particle is twice that of spheres. On the basis of shape factors, Heywood (27) has proposed a method for correcting equivalent diameters and equivalent velocities by means of plots of true Reynolds numbers *versus* Stokes-Reynolds numbers.

Since many of the methods described later have been used to measure the particle size of emulsions, it is desirable to know how liquids follow Stokes' Law. Bond and Newton (9) have shown theoretically and experimentally that surface tension is a factor to be considered in certain size ranges. Previously, Bond (8) had indicated that the terminal velocity of spherical drops or bubbles may be up 1.5 times that of solid spheres of the same shape and mass.

The preceding review of studies on the settling velocity of irregularly shaped particles of ideal configuration suggested a similar study on ground materials of various shapes. For this purpose the same five fractions were used on which the microscopic counts had been made. A sedimentation analysis was made by means of the Wagner turbidimeter (4), and a plot of the diameter (calculated from Stokes' Law) against the fractional transmission was drawn. From this plot, shown in figure 3, the mean sedimentation diameter was obtained by interpolation of the midpoint of the curve in the region of steep slope. The mean diameters are given in table 1, and a comparison of  $d_s$  and  $d_b$  is shown in table 2. The data indicate that the sedimentation diameter may be from 0.5 to 0.9 the



microscopic dimension and that the difference in the two diameters is less where the several dimensions of the individual particles are similar in magnitude.

The variation in the ratio of  $d_s/d_b$  explains the discrepancies noted in the literature where size distributions based on sedimentation methods are compared with microscopic counts. It is obvious that such comparisons cannot be made

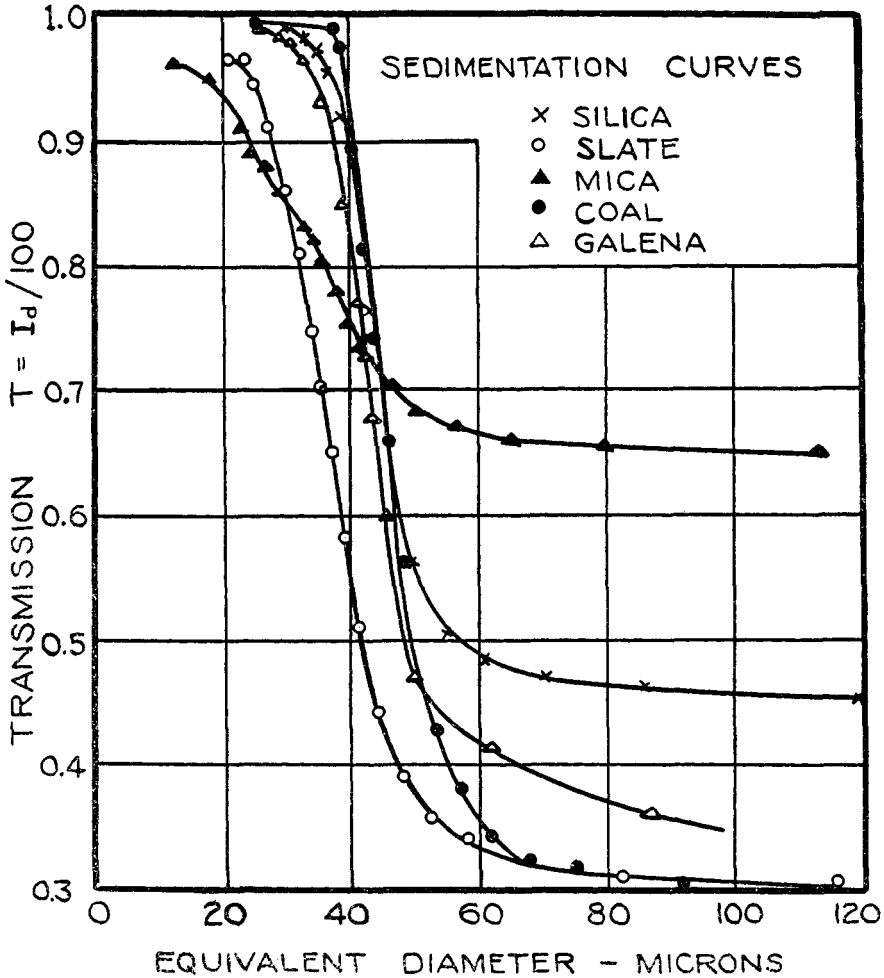


FIG. 3. Sedimentation curves

unless corrections are applied to one or the other diameters. In the case of mica the low ratio is a result of the effect discussed by Squires and Squires (41), who showed that the sedimentation velocity of thin discs when oriented vertically is 57 per cent greater than when oriented horizontally. Actually, a random orientation probably exists for platelike particles, giving a measure of particle-size distribution that is a "statistical" measure when sedimentation methods are used for such materials.

From a practical viewpoint the relation of settling diameter to sieve aperture is very important. Fritts (19) has shown that for a certain cement the diameter of spheres having the same velocity of settling as the particles passing a No. 325 sieve is 45 microns. A recent communication from him has indicated that for other cements the diameter of the sphere is more nearly 1.21 times the sieve aperture which is the mean of the diameter of the circumscribing circle and that of the circle inscribed within a square aperture. Other investigators have indicated a similar relation for different types of materials. The results for the five materials used in this study are given in table 2 and indicate some variation. The particle diameter may be either greater or smaller than the sieve aperture, depending upon the material. The relative settling velocities of the platy and acicular particles are smaller than for regularly shaped particles of the same diametric dimension, and, in addition, the settling diameter is smaller than the sieve aperture.

However, the size passing a No. 325 sieve from sedimentation analysis of the whole powders from which these fractions were obtained does not show the wide variations just discussed. If the size at the per cent passing a No. 325 sieve (corresponding to the amount obtained on the standard 1-g. wet test) is read from the cumulative per cent curve obtained by sedimentation analysis, it is found that the diameter is approximately the same within the limits of experimental error for most materials, as shown in table 1 under the  $d_h$  column. The distribution curves of the whole powders were averaged results from sedimentation analyses by pipet, hydrometer, and/or air elutriation. The value for  $d_h$  would be expected to be larger than  $d_s$  for the fractions discussed previously, since  $d_h$  in reality measures the upper limit of the particles just passing the sieve. The fact that  $d_h$  is approximately the same for different materials has a definite practical application where the particle-size distribution is such that both sieve analysis and subsieve methods must be used. An arithmetic average of all figures (except for mica) gives a value of 53 microns, which can be used as the point of separation for the No. 325 sieve where combined sieving and sedimentation methods are used. The data for mica are abnormal, as might be expected, since sieve analysis of platelike materials gives erratic results. This is true because the plates lie with their flat sides on the sieve, thereby tending to reduce the aperture.

The mean value of 53 microns is 1.21 times the sieve aperture, which is in accord with the results of Andreasen (3). In that study it was found (for flint particles only) that if the sedimentation diameter were multiplied by 0.806 (the ratio of the edge of a cube to the diameter of a sphere of the same volume), the sedimentation data would agree with sieve data. Thus, the sedimentation diameter was 1.22 times the sieve opening. This ratio (0.806), however, must not be inferred to apply in the conversion of sedimentation diameters to microscopic dimensions, since the latter are larger than the former, as has been discussed. It should be noted that in using these corrections for combined sieve and sedimentation analysis, it is more expedient to correct the sieve dimensions to equivalent sedimentation diameters than the reverse, because most calcula-

tions in practice are based on subsieve data, which are determined for the most part by sedimentation methods.

B. PARTICLE SHAPE

While there have been many published articles on the measurement of the properties of particles wherein the particles are assumed to be spheres, there are

TABLE 3  
*Variation in shape constant ratio for different geometric shapes*  
*a is length; b is dimension at right angles to a; c is thickness*

SHAPE	SURFACE	$f_s$	VOLUME	$f_v$	$K$
Disc: $a = b$ .....	$\pi b(b/2 + c)$		$\pi b^2 c/4$		
$c = 0.1b$ .....		$3\pi/5$		$\pi/40$	24
$c = 0.2b$ .....		$7\pi/10$		$\pi/20$	14
$c = 0.5b$ .....		$\pi$		$\pi/8$	8
$c = b$ .....		$3\pi/2$		$\pi/4$	6
Plate: $a = b$ .....	$2b(b + 2c)$		$b^2 c$		
$c = 0.1b$ .....		2.4		0.1	24
$c = 0.2b$ .....		2.8		0.2	14
$c = 0.5b$ .....		4		0.5	8
$c = b$ .....		6		1	6
Bar: $b = c$ .....	$2b(b + 2a)$		$ab^2$		
$a = 10b$ .....		42		10	4.2
$a = 5b$ .....		22		5	4.4
$a = 2b$ .....		10		2	5
$a = b$ .....		6		1	6
Cube: $a = b = c$ .....	$6b^2$	6	$b^3$	1	6
Sphere: $a = b = c$ .....	$\pi b^2$	$\pi$	$\pi b^3/6$	$\pi/6$	6
Tetrahedron:	$2.3b^2$	2.3	$0.18b^3$	0.18	13
$a = 1.16b = 1.23c$ .....					
Cone:	$0.81\pi b^2$		$\pi b^3/12$		
$a = b = c$ .....		$0.81\pi$		$\pi/12$	9.7
Double cone:	$1.12\pi b^2$		$\pi b^3/6$		
$a = 2b = 2c$ .....		$1.12\pi$		$\pi/6$	6.7

relatively few papers in which the authors have attempted to evaluate shape in absolute terms. Green (23, 24) was one of the first to discuss the necessity for considering the shape in relation to volume and surface. Heywood (26, 27, 28) has measured the surface and volume of relatively large particles. From these results he proposed shape factors based on the projected area of the particle and its thickness but, as might be expected, these relations become exceedingly complex in defining surface and volume. Furthermore, there is no

reason to assume that the relations for macroscopic particles apply to microscopic ones. It is quite possible that, as subdivision takes place, the particles tend to lose the shape of the mass from which they were derived and may or may not become spheroidal.

Wadell (48) has proposed a degree of sphericity which is the ratio of the surface of a sphere of diameter,  $d_n$ , to the actual surface of the particle and is equal to  $\sqrt{36/s}$ , where  $s$  is the area of a solid having unit volume but the same shape as the particle. When the particles are in the microscopic range, it appears that such a treatment is too difficult to use.

Dalla Valle (13) and Dalla Valle and Goldman (14) have studied the individual shape factors for silica, using the treatment of Hatch and Choate (25) for estimation of the mean size. The surface shape factor (the number required to convert the square of the mean diameter to actual surface) was determined from the pressure-drop relation of Burke and Plummer (10) for fluids passing through a column packed with the material. The volume shape factor (the number required to convert the cube of the mean diameter to actual volume) was determined by direct count of a weighed amount of particles. Their results

TABLE 4  
*Evaluation of shape constant ratios for various types of materials*

TYPE	FORM	EXAMPLES	K
Regular.....	Cubical	Galena	6-8
Angular.....	Tetrahedron	Silica, coal, cement	6-12
Acicular.....	Bar, cone	Slate, talc	4-10
Platelike.....	Plate, disc	Mica, talc	6-14

showed the surface shape factor to be less than  $\pi$  and the volume shape factor to be less than  $\pi/6$ . These values set as a maximum are those of a sphere and represent a convenient datum for studying shape factors but do not represent the maximum values, as shown in table 3.

The principal purpose in evaluating particle shape is for the determination of specific surface. As will be shown later, specific surface is directly proportional to the ratio of the surface shape factor,  $f_s$ , to the volume shape factor,  $f_v$ . If  $f_s$  and  $f_v$  are defined as the factors which relate the surface and volume of a particle to the square and cube, respectively, of its diameter, the problem is simplified and allows greater practical application of these functions than is possible with those proposed heretofore. If the shape constant ratio,  $K$ , is defined as the ratio of  $f_s/f_v$ , it can be shown readily that  $K$  does not vary greatly for various smooth geometric shapes. This is illustrated by the data in table 3, which show that the shape constant ratio lies between 4 and 14 for geometric shapes that are probably representative of the shapes approached by particulate materials. From a knowledge of the characteristics and microscopic dimensions of such particles it is then possible to assign values for their shape constant ratios to various types of materials. In this manner a more logical estimation

of specific surface may be made than to assume a value of 6 (for a sphere) in all cases. Such an estimation of shape constant ratios has been made in table 4.

The advantage of this method of treating the shape function is that the evaluation of one shape constant ratio eliminates the separate determination of the individual surface and volume constants. Furthermore, the shape constant ratios are not subject to as great variations as are  $f_s$  and  $f_v$  (see table 3). Values for  $K$  based on microscopic inspection of the dimensions of representative particles of the closely fractionated materials used in this study were estimated as given below, assuming a smooth surface:

MATERIAL	$K$
Silica, D-2.....	8
Slate, 470.....	7
Mica, 432.....	14
Coal, C.....	8
Galena, G.....	6

#### C. STRUCTURE

The surface configuration of a particle also has an important effect on its industrial use, since the structure may vary from the smooth surface of galena, figure 1F, to the porous structure of diatomaceous earth, figure 1H. The structure may exert its influence in the scattering of radiation (visible, infrared, etc.) to which it is exposed or in the actual surface exposed to chemical reactions. Structure is a difficult property to evaluate, and until its relations to other properties are first established it cannot be evaluated. The shape factor ratio,  $K$ , by definition includes both shape and structure considerations; this indicates that a combination of both these properties might be evaluated indirectly by such means as adsorption or light scattering.

#### D. SIZE DISTRIBUTION

The preceding discussion relates mainly to the study of the properties of individual particles; in industrial practice, however, the principal emphasis is on the properties of mixtures of particles either of a large size range or a rather closely sized fraction. Thus, the size distribution becomes a primary property in describing the characteristics of a powder, since it affects the secondary properties. The principal means of evaluating size distribution is to determine a weight distribution, i.e., to obtain the per cent by weight between certain size limits. From such a distribution the suitability of a material for a particular purpose may be ascertained by inspection of the size-distribution curve or by calculation of some property in quantitative terms, such as specific surface or a uniformity coefficient.

A plot of cumulative per cent undersize *versus* log diameter is a relatively simple means of comparing size-distribution data and allows rapid estimation of the adaptability of a material for a particular use. Such plots have been

used in comparing the weight distributions for the studies of microscopic diameters shown in figure 2. In addition, there are shown the size distributions obtained from sedimentation data for the same five materials. The turbidity data were converted to size distribution according to the method of Wagner (4). For these rather closely sized materials a constant transmittancy constant for all sizes, as used by Wagner, was assumed valid. With the closely fractionated materials used in this study the theoretical treatment of Wagner appears valid, on the basis of the results for  $d_s$  and  $d_t$  shown in table 1.

Other graphical methods for showing size distribution have been proposed, and a review of them has been given by Austin (6). This author presented the mathematical relations for evaluating size distribution and recommended the use of log probability curves. For many materials the data approximate straight lines, but this treatment tends to desensitize the data to a considerable extent. In addition, these plots will not apply rigorously to the size distributions of certain materials, depending upon the operations used in their production. In such cases a cumulative per cent undersize *versus* log diameter plot<sup>2</sup> will show the correct size distribution and a frequency-log diameter curve, such as per cent per micron *versus* log diameter, will be still more sensitive to slight variations in distribution. Heywood (27) has proposed a modified frequency plot in which the coördinates are corrected for mean size, thus allowing materials of considerable difference in size to be plotted on the same chart for comparisons of size distribution. Work (56) has proposed a frequency-diameter plot consisting of a primary and secondary relation which can be reduced to a single equation. Roller (39) has proposed a similar type of plot. Both these equations are more complex than the log probability types, but significant peaks in the distribution are not smoothed by desensitization. Vieweg (47) has suggested a method of evaluation based on the reciprocal of the fourth power of the diameter.

The degree of precision of the size measurement is the limiting factor in the use of sensitive frequency distribution plots and consideration must be given to the use of the plot for interpretation of the results before any one type of curve is selected. Thus, for control work it is necessary that a rapid evaluation of the size distribution be obtained, and complex plots are not suitable.

Other methods of quantitatively evaluating size distribution are those in which some mean diameter is calculated, such as a surface mean diameter,  $D_s$ :

$$D_s = 100/(\sum G_f/d_m), \text{ microns} \quad (3)$$

where  $G_f$  is the weight per cent in the individual fractions, and  $d_m$  is the arithmetic mean of the limits of the fractions, in microns.

In a similar manner a weight mean diameter,  $D_w$ , may be obtained from:

$$D_w = (\sum G_f d_m)/100, \text{ microns} \quad (4)$$

<sup>2</sup> In drawing the curve for a cumulative per cent curve, the nature of the curve demands that all points be connected rather than that a smooth curve be drawn between all points, if the precision of the method warrants it.

It can be shown by experiment that  $D_w$  from equation 4 is the size at which there is 63 per cent by weight finer for a wide variety of materials. The size at 63 per cent finer is a parameter of the Rosin-Rammler equation, as given by Austin (6). Since it is a distribution constant, it probably can be derived from equation 4 and the Rosin-Rammler law. This has been attempted without

TABLE 5  
Comparison of  $D_w$  and  $d_{63}$

MATERIAL	WEIGHT MEAN DIAMETER $D_w$	SIZE AT 63 PER CENT FINER $d_{63}$
	<i>microns</i>	<i>microns</i>
Jaw-crusher feed		
Traprock.....	7,750	7,300
Limestone.....	21,200	20,900
Jaw-crusher product (classified)		
Limestone.....	11,800	11,200
Mica schist.....	9,400	8,400
Roll-mill product (classified)		
Limestone.....	3,430	3,150
Ball-mill product		
Traprock (1 hr.).....	281	65
Mica schist (1 hr.).....	159	150
Limestone, 957	55	50
Plus 40-micron material.....	100	110
Minus 40-micron material.....	16	19
Red slate, 642.....	11	13
5-10 micron fraction.....	9	10
20-40 micron fraction.....	38	31
Green slate, 470.....	17	12
Traprock, 113.....	25	30
Pumice, 571.....	11	10
Green mica, 432.....	18	17
Talc, 573.....	18	19
Diatoms, 939.....	13	11
Silica, 196.....	21	22

success, although Hottel and Stewart (29) have derived an analogous relation for studies on coal. Experimental data are given in table 5, comparing  $D_w$  and the size at 63 per cent finer. With the exception of one or two materials, the results are in good agreement, indicating the general applicability of selecting the mean size at 63 per cent passing without recourse to computation by equation 4. In the case of the ball-mill traprock ground for 1 hr. the results do not agree, but neither is the Rosin-Rammler plot a straight line. This is to be

expected if the effects of the size distribution of the feed are not eliminated by sufficient grinding. The feed effects can be illustrated by a frequency plot of such materials which will show a double peak. The peak in the coarse size range has a great effect on the weight mean diameter, and, accordingly,  $D_w$  is not equal to the size at which there is 63 per cent undersize. The type of material being ground also has an effect on the time required to reduce the peak in the coarse size range to a normal distribution, as is shown by the results for the mica schist ball-mill product ground for the same time. The feed size of the traprock was minus a No. 6 sieve and plus a No. 35 sieve, while that for the mica schist was minus a No. 8 sieve and plus a No. 35 sieve.

It can be shown readily that an infinite number of size distributions may give the same numerical values for either of the two mean diameters given by equations 3 and 4. However, in evaluating the properties of a series of materials produced in the same manner, these mean diameters are satisfactory to use if the same fraction limits are applied in each case.

A finely divided material may be further described by computing the uniformity, i.e., the range of the property compared to the mean. Briefly, these relations may be computed from the equation

$$A = \sqrt{\frac{(D - d_m)^2 F_f}{\Sigma F_f}} \quad (5)$$

where  $A$  = the standard deviation,

$D$  = the mean diameter of the whole powder based on  $F$ ,

$d_m$  = the mean diameter of each fraction, and

$F_f$  = the value of the property for each fraction and may be number, surface, or weight.

The deviation can then be corrected for actual size (24) by computing the uniformity coefficient,  $U$ , where the terms are the same as in equation 5:

$$U = D/(A\sqrt{2}) = 0.71D/A \quad (6)$$

Certain other arbitrary methods are used to evaluate the spread of sizes, such as the uniformity coefficient used for filtration sands:

$$U = d_{60}/d_{10} \quad (7)$$

where  $d_{60}$  and  $d_{10}$  are the sizes which show 60 and 10 per cent finer, respectively, as interpolated from a cumulative per cent *versus* diameter curve.

The final choice in selecting methods for evaluating size distribution is dependent upon the use to be made of such information. The most expedient ones to employ are those that yield the necessary information to differentiate materials the properties of which are similar in other respects but which behave differently when used in a particular product.

## II. SECONDARY PROPERTIES

### A. SURFACE

The surface of a particle or of a group of particles is dependent upon the primary properties previously discussed. In general, it is evaluated for groups of particles having a more or less wide range of sizes.



The surface of the particles can best be defined as the specific surface,  $\underline{S}$ , which is the surface possessed by a unit weight and is generally expressed as square centimeters per gram. It may be calculated by means of the following equation (cf. equation 3):

$$\underline{S} = \Sigma S_f = \Sigma \frac{KG_f \times 10^2}{D_s \times d_m} = \frac{K \times 10^2}{D_s} \Sigma G_f/d_m = \frac{K \times 10^4}{D_s \times D_s} \quad (8)$$

where  $\underline{S}_f$  = the surface in the individual fractions per gram of whole powder,  
 $K$  = the shape ratio constant ( $f_s/f_v$ ),  
 $G_f$  = the weight per cent in the individual fractions,  
 $D_s$  = the density of the material, in grams per cubic centimeter,  
 $D_s$  = the surface mean diameter (equation 3), in microns, and  
 $d_m$  = the arithmetic mean diameter of the limits of the fraction, in microns.

The above equation assumes that the mean particle size of the fraction is at the midpoint of the fraction; this is a valid assumption provided the spread of sizes is not very great in the small size ranges. It is an expedient treatment to use for surface computations where the weight distribution is known. However, it requires knowledge of the ratio of the surface-to-volume shape constant,  $K$ . In most published work, this constant has been assumed as  $\pi/(\pi/6)$ , or 6, which is the value for a sphere, but in the approach to absolute evaluation of specific surface it is necessary to assign more accurate values to this constant for different types of materials.

One of the principal uses of the per cent per micron *versus* log diameter plot is for the estimation of surface, since the area under such a curve is directly proportional to specific surface, as was indicated by Roller (38).

If  $G$  is cumulative weight per cent and  $\Delta G$  is the weight per cent between two diameters,  $\Delta D$ , then the per cent per micron is  $\Delta G/\Delta D$  and the area,  $A$ , under the curve is

$$A = \int dA = \int (dG/dD) \cdot d(\log D) = (1/2.3) \int (dG/dD) \cdot (dD/D) \\ = (1/2.3) \int dG/D \quad (9)$$

The volume of a particle is  $f_v D^3$ . Its weight,  $W_f$ , is  $f_v D^3 D_s$ , if  $D_s$  is the density. Its surface is  $f_s D^2$ . The surface, therefore, in square centimeters for  $D$  in microns is given by

$$S_f = \frac{f_s W_f \times 10^4}{f_v D_s \times D} = \frac{K W_f \times 10^4}{D_s \times D} \quad (10)$$

where  $K$  is the shape constant ratio,  $f_s/f_v$ .

The specific surface,  $\underline{S}$ , in square centimeters per gram is equal to the sum of the individual surface from each fraction given by equation 10 divided by the total weight of the material, or

$$\underline{S} = \Sigma S_f / \Sigma W_f = \frac{K \times 10^4}{D_s \times \Sigma W_f} \times \Sigma W_f / D \quad (11)$$

But  $(W_f/\Sigma W_f) \times 100$  equals  $\Delta G$ ; therefore

$$\underline{S} = \frac{K \times 10^2}{D_s} \times \Sigma \Delta G/D = \frac{K \times 10^2}{D_s} \int dG/D \quad (12)$$

or

$$\underline{S} = \frac{2.3 \times K \times 10^2 \times A}{D_s} \quad (13)$$

The numerical value of  $A$  may be obtained by means of a planimeter or by the more approximate graphical integration

$$A = \Delta(\Delta G/\Delta D) \times (\log D)_a \quad (14)$$

where  $\Delta G/\Delta D$  is the per cent per micron at  $(\log D)_a$ , the latter being the arithmetic average of the logs of the limiting sizes of the fractions.

The specific surface as computed by equation 13 is somewhat more accurate than the results from equation 8, since inflections in the size-distribution curve are magnified. For the same reason the use of equation 13 necessitates methods for determining size distribution that yield close fractionation with a higher degree of precision. In practical applications, equation 8 is recommended because, in general, the methods for measuring particle-size distribution that are available lack the precision required for equation 13 and, in addition, the use of equation 8 is less laborious. Another graphical method for obtaining the specific surface is by means of the Gates diagram (21), which is a plot of cumulative per cent finer *versus* the reciprocal of the diameter. It can be shown readily that the area under such a curve is proportional to surface, since the area equals  $\int_0^{100} dG/D$ .

In addition to the above methods of determining surface from weight size distributions, means have been proposed for estimating the gross surface of the powder other than from the surface of the individual fractions. Emmett and coworkers (17) have evaluated surface by adsorption isotherm data which, when compared with results from hydrometer analysis on soils, gave "a little higher results." Askey and Feachem (5) have used a similar method. Other methods for estimating specific surface from permeability data have been proposed and will be discussed under packing properties. For other indirect methods used to estimate surface, reference should be made to the bibliographies of Roller (40), Work (55), and Heywood (27). Such methods do not yield information regarding the size distribution, as actually an infinite number of distributions may yield the same specific surface.

A computation of specific surface by means of equation 8 was made for the five closely fractionated materials studied previously, wherein  $G_f$  was made equal to 100 and  $d_m$  was the measured mean diameter. The results are shown in table 1, where the necessity for standardized procedures for evaluating particle size and shape is evident. Thus, the relative surface shown by silica and mica will be considerably different, depending upon whether settling diameter or microscopic diameter is used as the criterion. Furthermore, their relative sur-

face based on any given diameter will depend upon the value of  $K$  used. If different types of particles can be classified as to their general shapes, it will then be possible to evaluate  $K$  and in turn the specific surface in terms which will be an approach to absolute values.

#### B. PACKING PROPERTIES

A secondary property that is used to a considerable extent in industry for evaluating finely divided materials is their packing or bulking properties. Such evaluation may take one of a number of forms. The bulk density,  $B_c$ , may be defined as the ratio of the volume (particles plus voids) occupied by a unit weight of the material. Such determinations when used for comparison should be made under the same conditions to get the same degree of compaction. A simple method of determining the bulk density consists in filling a 100-cc. graduate to the top with the dried material and then compacting it by allowing the graduate and contents to bounce on a rubber-covered brick. These operations are continued until the volume no longer decreases. In general, this end point is denoted by a definite change in the sound created by the compacting operation. Such a compaction may be considered to be "to completion," and from the observed volume and the weight of the material the bulk density and per cent voids may be calculated. For obtaining the compacted weight of materials in the loosely compacted state, the Scott volumeter is suggested. In this apparatus the powder is allowed to flow at a uniform velocity into a small box of known volume. From the weight of the powder the bulk density,  $B_l$ , can be computed for the loose condition of packing.

In order to compare materials of different true densities,  $D_s$ , it is expedient to calculate the per cent voids in the compacted mass. The per cent voids,  $V$ , may be calculated from the bulk density by means of the following equation:

$$V = 100(1 - B/D_s) \quad (15)$$

Traxler and coworkers (44) give a discussion of the packing of mineral powders, as do also Work (55), Roller (37), and Burmister (11). These methods are suggested for use only as approximations in evaluating relative sizes, since size distribution and particle shape have a considerable effect on the results. The effect of size distribution is used by Anderegg (1) in selecting gradings for desired densities. The mathematics of the packing of spheres has been discussed by White and Walton (54) and by Manegold and coworkers (31, 32), but the application to irregular shapes is quite complex. These packing properties have been used to a considerable extent in evaluating the properties of fillers used for asphalt, in studies of aggregates used for concrete, and in powder metallurgy.

A more elaborate test of compacted mineral powders consists in the evaluation of the permeability of the compacted mass from pressure drop relations. Traxler and Baum (45) determined the average void diameter for different ground minerals and indicated that it was related to the average particle size of the material. A compilation of such data and other properties of various

TABLE 6  
*Typical particle-size data on various materials*

MATERIAL	NO.	$D_s$	$D_s^*$	$D_w$	$B_c$	$V$	$d_v$
		grams per cc.	microns	microns	grams per cc.	per cent	microns
Limestone.....	107	2.73	6.4	29	1.78	34.6	1.14
Traprock.....	113	2.91	6.3	25	1.58	45.6	1.63
Coal.....	C	1.75	9.8		1.18	33.0	
Galena.....	G	7.66	15		4.94	35.6	
Glass spheres†.....	S1	2.50	127	129	1.48	40.8	40.6
Glass spheres‡.....	S2	2.50	94	95	1.49	40.3	29.2
Mixed glass spheres.....		2.50	133		1.59	36.2	34.7
Asbestos.....	38	2.61			0.92	65.0	
Zinc powder§.....	2196	7.07			4.16	41.1	2.46
Aluminum powder§.....		2.70			0.75	72.5	
Titanium dioxide§.....	TO	4.10			0.72	82.5	
Silica.....	196	2.62	5.5	21	1.64	37.5	1.07
0-2 microns.....		2.62	1.2		0.92	65.2	1.01
2-5 microns.....		2.62	3.9	4.2	1.24	52.7	1.20
5-10 microns.....		2.62	7.7	8.4	1.37	47.5	2.07
10-20 microns.....		2.62	15	16	1.47	44.7	2.85
20-40 microns.....		2.62	31	33	1.45	43.7	6.02
40-80 microns.....		2.62	59	61	1.44	44.1	9.08
Black slate.....	197	2.91	5.1	9.9	1.24	57.4	1.66
0-2 microns.....		2.91	1.0		0.84	71.8	1.01
2-5 microns.....		2.91	4.8	5.1	0.87	70.3	1.16
5-10 microns.....		2.91	7.5	7.6	0.97	66.6	1.72
10-20 microns.....		2.91	12	12	1.06	63.6	2.52
20-40 microns.....		2.91	18	19	1.14	60.9	3.85
40-80 microns.....		2.91	44	45	1.32	54.8	7.00
Red slate.....	642	2.80	2.2	11	1.30	53.4	0.87
5-10 microns.....		2.80	8.1	9.3	1.49	46.7	2.26
20-40 microns.....		2.80	34	38	1.54	45.2	6.64
Green slate.....	470	2.82	3.9	17	1.21	57.0	1.27
Green slate.....	471	2.82	3.2		1.02	63.8	1.30
Cement.....	2135L	3.08	6.7¶	13.1	1.44	53.3	1.83
Cement.....	2194NA	3.14	7.4¶	16.3	1.58	49.7	1.58
Green mica.....	432	2.92	9.2¶	18	0.89	69.5	1.30
Diatoms.....	939	2.19	4.8¶	13	0.48	78.3	2.63
Talc.....	573	2.82	10.8¶	18	1.12	60.2	1.31
Pumice.....	571	2.38	5.7¶	11	1.00	58.0	1.66
Tripoli.....	223	2.60	7.8¶		1.20	53.7	2.04

\* Data for fractions of 196, 197, 642 and glass spheres are based on microscopic counts; others on sedimentation analysis.

† Minus No. 100 sieve plus No. 140 sieve.

‡ Minus No. 140 sieve plus No. 200 sieve.

§ Commercial product.

¶ Air-elutriation data.

materials is given in table 6. The fractions of whole powders were obtained by means of a Federal pneumatic separator (18).

The calculation of specific surface (or average particle size) from permeability data has received considerable attention recently, because of the speed with which the results may be obtained. These methods are based on an empirical relation proposed by Carman (12) for relating surface and pressure drop through packed solids. The results for the specific surface of silica (5420 sq. cm. per gram) and of black slate (7040), calculated from permeability data of Traxler and Baum (40), are considerably higher than similar results (4150 and 4050, respectively) computed from sedimentation data assuming spherical shape with  $K$  equal to 6. Lea and Nurse (30) have proposed a similar method for evaluating specific surface based on Carman's work but using gases instead of liquids, as was done by the latter. These investigators indicated that liquids tend to give high results, although their values using air for cement and ground sand were about 20 per cent higher than the results computed from size-distribution data obtained by pipet analysis. By using Andreasen's relation (discussed under sedimentation diameter), the authors showed that the results by the two methods could be made to agree by multiplying the settling diameter by 0.806. The net result of this correction is to evaluate specific surface in terms of a diameter reduced to a hypothetical sieve dimension equivalent to the settling diameter. Such a basis for surface evaluation is not tenable, since, as was illustrated in table 1 and as has been shown by others, all data point to the fact that the actual size is greater than the sieve dimension. In most cases permeability surface tends to give higher results (42) than specific surface results calculated from size-distribution data obtained by other methods. The variations are caused by considerations of the shape constant ratio,  $K$ , and the values used for the Carman empirical constant in converting permeability data to surface.

In connection with permeability measurements, it should be pointed out that a gross size function only is measured, and no data are obtained on size distribution. These empirical methods of Carman, Lea and Nurse, and Gooden and Smith (22), all of which are similar, may apply for selected powders, but they cannot be used with confidence until the relations between packing (a secondary property) and the primary properties are rigorously established. Permeability analysis would appear to offer interesting possibilities in evaluating particle shape and the rapid estimation of specific surface for control purposes. Such methods are particularly adaptable to studies of mixtures of materials of different densities, which otherwise require microscopic counts or special apparatus and techniques.

### III. CONCLUSIONS

From the results of this investigation the following conclusions were drawn:

1. For many materials, the  $d_b$  diameter (shorter of the two visible microscopic dimensions) yields essentially the same results as the  $d_k$  diameter (square root of the projected area). An exception was micaceous material, for which the former gives lower results; for the particular mica used in this study the ratio of  $d_b/d_k$  was about 0.9.

2. On the basis of the results for a No. 325 sieve (nominal aperture 44 microns), the mean  $d_b$  dimension of particles just passing may vary from 1.2 to 1.5 times the aperture.

3. The mean sedimentation diameter (diameter of the equivalent sphere) varies from 0.5 to 0.9 of the  $d_b$  dimension, depending upon the material. This variation accounts for the discrepancies between the results for particle-size distribution obtained by microscopic counts and those from sedimentation methods.

4. While the mean sedimentation diameter of particles just passing a No. 325 sieve may vary from 0.8 to 1.2 times the sieve aperture, the separation point for most materials may be taken as 53 microns or 1.2 times the aperture, since the point of separation in reality measures the upper limit of the material just passing. A single sieve correction factor (1.2 times the aperture) may be used expediently for all materials for which combined sieve and sedimentation analyses are employed, in order to put the entire analysis on a sedimentation diameter basis.

5. In practice it is not necessary to determine individual surface and volume shape factors if the shape constant ratio is known, since the latter is sufficient for the computation of specific surface from weight distribution.

6. The average weight diameter,  $D_w$ , is equal to the size at which there is 63 per cent undersize for many pulverized materials, indicating that this size, which is a parameter of the Rosin-Rammler law, is a significant property of such materials.

7. Empirical methods for evaluating specific surface from permeability data cannot be used with confidence until the basic relations between packing (a secondary property) and the primary properties are established.

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