

# Glass Spheres for the Measurement of the Effective Opening of Testing Sieves<sup>1</sup>

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It is shown that the "effective" opening of testing sieves is generally somewhat larger than the average opening or the nominal opening. A method of measuring the effective opening by means of a calibrated mixture of glass spheres is described. Glass beads of the type used for highway markings were a suitable source of glass spheres for sizes from 80 to 1,000 microns (U. S. Sieves No. 170 through 18). Details are given for the selection of the spheres, the preparation of the desired mixture of spheres, subdivision of the samples, the measurement of the diameters, and the calculation of the weight-size distribution. From a statistical analysis of the calibration data, both the reproducibility and accuracy of the method were found in general to be about 1 percent.

## 1. Introduction

The specifications for practically every powdered or granular material of commerce prescribe maximum and/or minimum *limits* of the particle sizes. In addition, a knowledge of the *distribution* of particle sizes is often required, both for control purposes and in research. In order to test materials for conformity with specifications, or to measure the particle size distribution, testing sieves are used by many industries. Sieve analyses obtained by different laboratories (for example, those of the buyer and seller) often show considerable disagreement. The discrepancy in some cases is due to inadequate sampling procedures, but when good representative samples, or even the same sample are used, differences frequently occur.

The sieving operation consists of agitating the material on a sieve of known opening until substantially all the particles that are small enough to pass the openings have passed. The shaking process can be carried out in a satisfactorily reproducible manner so that the error from this source can be made satisfactorily low [1].<sup>3</sup> The weights of the fractions retained by the various sieves can be determined without appreciable errors. On the other hand, the evaluation of the size of the openings of testing sieves may be one of the largest sources of error in sieve analysis. Consideration of this error is the subject of this paper.

Specifications [2] for testing sieves allow a manufacturing tolerance in the average size of the opening from  $\pm 2$  to  $\pm 7$  percent, depending upon the size of the opening. These tolerances are rather wide but are satisfactory if only a rough idea of the particle size is desired or if it is desired that a certain material be finer or coarser than some stated size. If an accurate particle size distribution is required, especially with material with a narrow range of particle sizes, then these tolerances are so large as to render the sieves almost useless without an individual calibration to determine the effective opening of each sieve.

### 1.1. Effective Sieve Opening

The difficulties encountered in the evaluation of sieve openings arise from three sources, none of which appear to have received sufficient attention. First, all the openings of a sieve are rarely of the same size. Rather, the sizes of openings are distributed according to some probability law. The over-sized particles can pass the larger holes and, thus, the openings that are effective are somewhat larger than the average opening. Second, the separation achieved by a sieve is not complete, but instead some under-sized particles always remain on the top of the sieve. The sieve opening is thus effectively somewhat smaller than the average opening. Third, the opening is effective in three dimensions, and the plane defined by the sieve cloth may not coincide with the plane defined by the effective opening. The effective opening will thus be larger than the average opening, which is measured by the projection on the plane of the sieve cloth.

The first and second of these phenomena are acting in opposite directions, and the effects of one might nullify the effects of the other. However, the manner and speed of shaking affect both; therefore, the effective opening is not a constant for a given sieve but depends also upon the way the sieve is used. The general dependence of the effective sieve opening on time of shaking is illustrated in figure 1. Assume that a sieve with an opening size distribution given in figure 1, A, is to be used to separate into two fractions a material with the particle size distribution given in figure 1, B. After a very short time of shaking, the particle size distributions in the fractions passing and retained are as illustrated in figure 1, C. The separation is very incomplete with large amounts of under-sized particles retained. After a longer shaking time the distributions given in figure 1, D, may be achieved, and the effective size of the sieve may be defined as the point at which the curves cross, which is also the point at which the curves are the steepest. Figure 1, E, illustrates the effect of an infinitely long shaking time. As the shaking progresses it is seen that the effective opening becomes larger. It becomes evident that the effective opening depends upon the time of shaking. In addition, the type of shaking motion would have a similar effect. Any method of calibration for effec-

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<sup>3</sup> Figures in brackets indicate the literature references at the end of this paper.

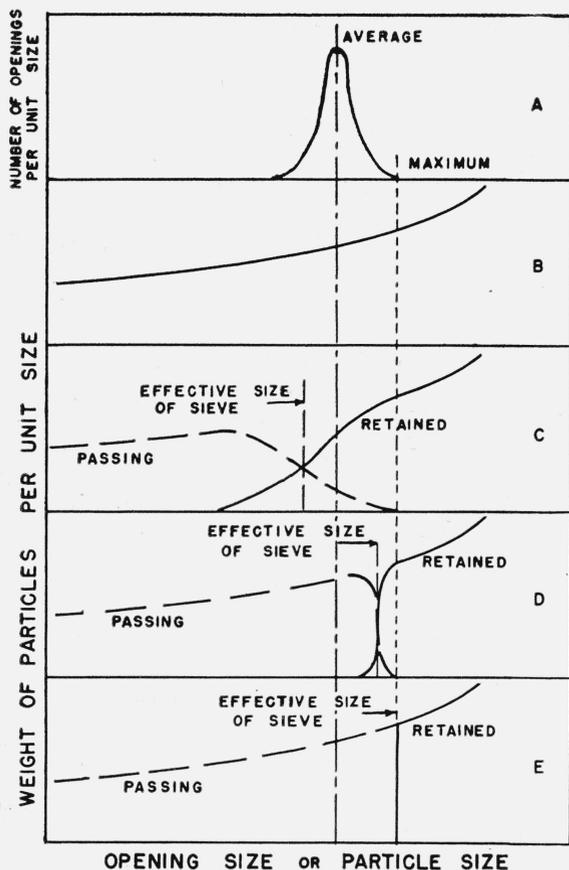


FIGURE 1. Effect of time of shaking on the effective sieve opening.

A, Opening-size distribution; B, particle-size distribution before sieving; C, particle-size distribution after very short time of shaking; D, particle-size distribution after long time of shaking; E, particle-size distribution after infinite time of shaking.

tive opening must necessarily take these two effects into account.

If all the openings of the sieve were exactly the same size, the shaking time required to reach a condition similar to figure 1, D, would be considerably shortened. It is to be noted, however, that even in perfectly uniform sieves the effective opening is not equal to the average opening unless the shaking is continued for an infinite time.

The usual method of calibrating sieve openings [3] consists in making measurements on a magnified projection of the sieve cloth. Observations by this method are rapid, and the openings may be inspected for uniformity during these measurements. This method, however, has two serious disadvantages. First, the average opening is measured, taking no account of the difference between average and effective opening. Second, the calibration requires special equipment and skilled operators. Weber and Moran [4] suggested that the projection method be used to measure a large number of openings, and the effective opening be determined by use of an empirical relation between the statistical parameters and the effective opening. This method requires

the same special equipment and even more skilled operators.

Fagerholt [5] showed that the effective size of the sieve after a shaking period of time,  $t$ , is equal to the average diameter of particles that passed by continued shaking under the same conditions during the interval of time  $t$  to  $3t$ . This conclusion is based on assumptions that are not strictly true. For practical purposes this method is much too involved, because it requires an independent particle-size determination for each sieve for each sieve analysis. It offers a way, however, for determining the effective size of the sieve for very irregularly shaped particles.

## 2. New Method of Calibrating Testing Sieves

The authors in a previous paper [1] pointed out that the effective opening can be determined directly and simply by measuring the sizes of spheres that will just pass. In that paper exploratory work was described on the use of a calibrated sample of glass spheres for measuring the effective opening of testing sieves.

### 2.1. Particle Shapes

It is recognized that the sieve openings are square or slightly rectangular in shape and that irregularly shaped particles can pass through even though one of the dimensions of the particle, or "an average" of all dimensions, is considerably larger than the diameter of the opening. This is especially true for needle-like shapes. The average diameter of irregular particles that pass a sieve cannot be considered equal to the diameter of spheres that pass the sieve. The glass spheres used in this work were only intended to measure the opening of the sieve. The calculation of some "average" diameter of particles, which deviate from a spherical shape, is a separate problem that introduces factors not directly related to the methods for evaluating effective sieve openings.

### 2.2. Rectangular Openings

If the openings are rectangular rather than square, then the "effective" size for irregularly shaped particles will be increased, but the effective size for spheres will be the same as for a square with the same dimension as the minimum side. For this reason, sieves that are to be used with needle-like particles must be examined independently for squareness of holes. Fortunately, the openings of most testing sieves made in recent years are essentially square in shape.

### 2.3. Uniformity of Openings

The method of calibrating testing sieves by use of glass spheres takes into account the effect of non-uniformity, but it does not measure directly the uniformity of the openings. If there is reason to suspect that the sieve openings are not sufficiently uniform, they should be checked independently either by the usual projection method or by the method proposed by the authors in a previous paper [1]. The latter

is based on the effect of uniformity of openings on the sieving rate. The amount of nonuniformity that can be tolerated in testing sieves was considered in [1]. Sieves that do not appear obviously deformed, are usually sufficiently uniform so that the glass sphere method of calibration will correct for the small amount of nonuniformity that does exist.

### 3. Selection of Glass Spheres

The spherical shape was chosen primarily to eliminate any doubt or question about "average" diameter or the orientation of the particle in passing the opening. The spheres were not intended to be representative of the particles to be used on the sieves, nor is their diameter to be considered equal to the average diameter of irregular particles that might be sieved on the same sieve.

Glass was chosen because its hardness minimizes abrasion and because spheres of glass are readily available. Glass beads of the type used for projection screens were used by Gooden and Updike [7] as a source for spheres of 100- $\mu$  diameter. Glass beads of the type used for highway markings<sup>4</sup> are a suitable source of glass spheres for sizes from 80 to 1,000  $\mu$  (sieves No. 170 through 18). Larger and smaller sizes of glass spheres are being made for other purposes, and a wider range of sieve sizes could be covered if desired.

#### 3.1. Separation of Spherical Particles

The glass beads for highway markings as purchased are not all spherical, nor are the sizes continuous. It was necessary to separate the spherical particles from the elliptical, tear drop, dumbbell, and jagged pieces. The spherical shapes were effectively separated from the other shapes by rolling the particles down an incline. The spheres roll true, whereas nonspheres either roll in circles or not at all. The apparatus used is shown in figure 2. It consists essentially of a smooth flat disk about 20 in. in diameter, turning at about 3 rpm and tilted slightly. Only one sieve fraction can be separated at a time. For the larger sizes (about 1,000  $\mu$ ) the inclination of the disk is only a few degrees from the horizontal, but for the finer sizes (100 $\mu$ ) inclinations up to 30° from the horizontal are required. It is desirable that the particles roll over as many times as possible; therefore, a disk 30 or 40 in. in diameter would be desirable for particles larger than 1,000  $\mu$ . For the larger spheres, a disk made of ordinary window glass is an acceptable material for the separation. No difficulty is experienced in cutting it to the desired shape, as it need not be perfectly round. It can be fastened to the turntable by means of a very viscous stopcock grease. For the smaller particles, glass has a serious disadvantage in that it rapidly builds up static electric charges. A polished copper plate is generally more satisfactory, however, and in these experiments a polished copper plate

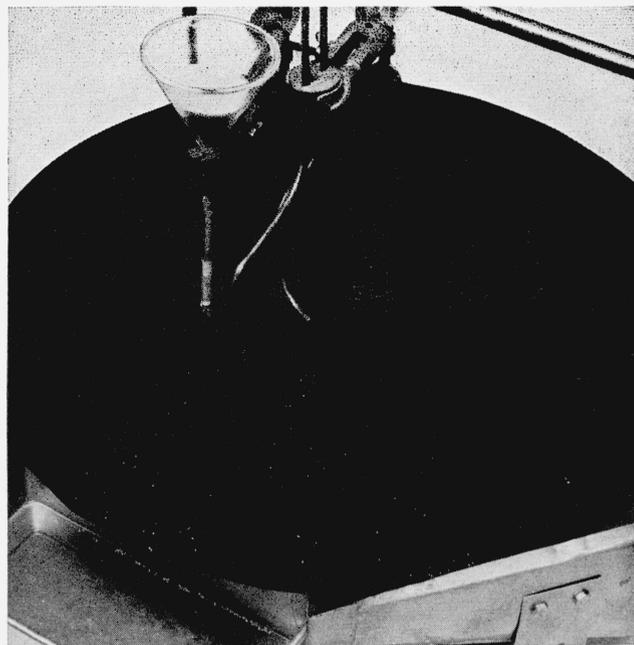


FIGURE 2. Apparatus employed to separate spheres from the original mixture.

one-sixteenth in. thick was used on particles smaller than about 500  $\mu$ .

The particles were fed onto the rotating disk in a thin stream. For the finest sizes the number rate of feed was quite large, and a 2-mm glass stopcock made a suitable gate. For the larger sizes only three or four pieces at a time could be fed without undue interference. A stopcock is unsuited at this low rate, and instead a cardboard tube with a notch in one side was supported to just touch the surface of the plate. The motion of the plate beneath the tube caused the particles to roll out of the notch in the tube one layer high and three or four wide (depending on the width and height of the notch). A gentle blast of air was directed into the stream of particles immediately after they were fed onto the plate. This dislodged any that stuck behind a nonspherical piece and started all particles rolling.

The spherical particles immediately rolled off the plate into a suitable hopper, while the nonspherical particles either did not roll at all or rolled more slowly or in curved paths and were carried by the turning disk and fell into the discard hopper. A strong blast of air was used to dislodge the particles that did not roll. Figure 3 shows the spherical particles, and figure 4 the discarded particles. About 10 lb of 74 to 149  $\mu$  (No. 100 to No. 200 sieves) material was separated in 20 hours on the apparatus described. About 5 percent of this particular material was discarded. In some lots, particularly in the larger sizes, 50 percent was discarded. The larger sizes separated more quickly.

Measurements of the sphere diameters showed that on the average the major and minor axes of the projections of the spheres differed by more than 2 or 3 percent only in 4 percent of the beads. Only one

<sup>4</sup> Suitable beads have been obtained from: Cataphote Corp., Toledo, Ohio; Potters Bros., Inc., Ozone Park, N. Y.; Minnesota Mining & Manufacturing Co., St. Paul, Minn.

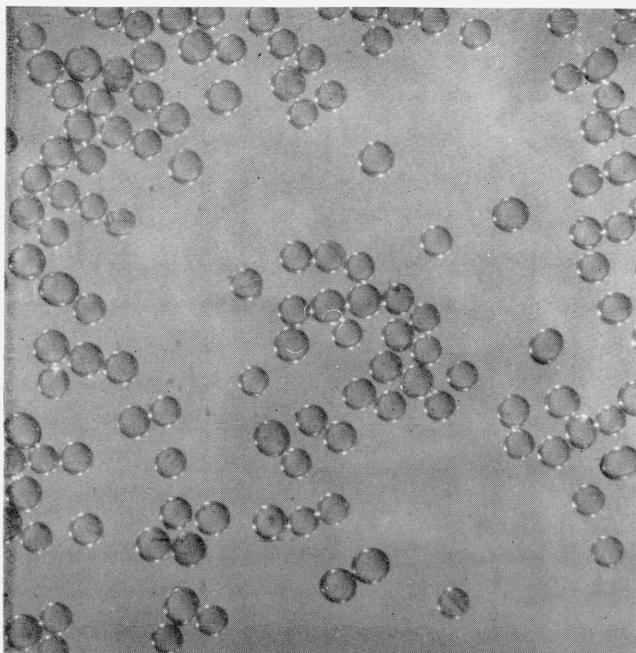


FIGURE 3. *Spherical particles separated from mixture.*

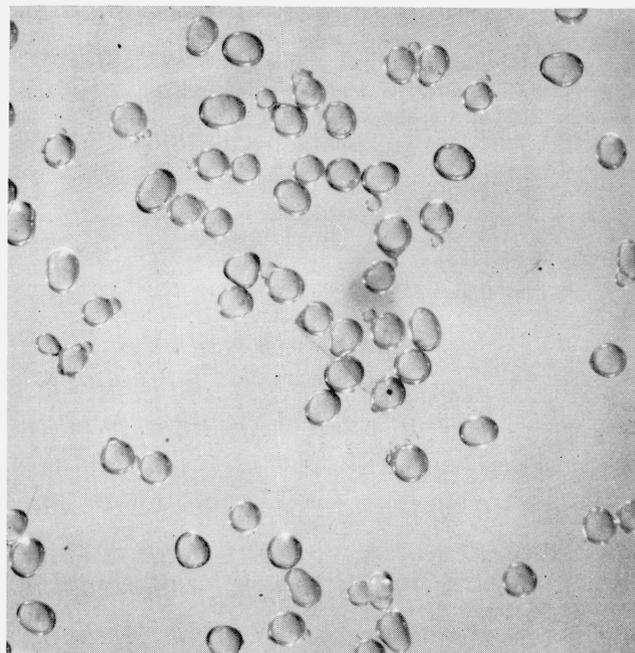


FIGURE 4. *Discard particles separated from mixture.*

bead in 14,000 was observed whose major axis was more than 1.5 times the minor axis. At least 90 percent of the beads could be considered spherical within the accuracy of the measurements.

### 3.2. Continuous Size Distribution

In order to obtain spheres of a continuous size distribution the material selected as spheres was carefully sieved into the closest sieve fractions ( $\sqrt[4]{2}$  series), and then equal weights of each fraction were used in the final mixture. This procedure produces a logarithmic particle size distribution. This distribution was chosen because the precision of the measurement of the sieve openings is the same for all sizes.

The details of the manufacturing process for these glass beads were not known. Various lots as received from the manufacturers were found to contain a discontinuous distribution of particle sizes. It is not known whether this resulted from the manufacturing process or from a subsequent particle size separation. For example, one lot of spheres was examined in which the 350- to 500- $\mu$  sieve fraction (No. 45 to No. 35) consisted entirely of spheres ranging from 350 to 400  $\mu$  and from 440 to 500  $\mu$ . There were no spheres at all in the size range 400 to 440  $\mu$ . The No. 40 sieve has a nominal opening of 420  $\mu$ , and this lot consisted of equal weights of No. 35 to No. 40 fraction and No. 40 to No. 45 fraction. The sieves indicated a smooth continuous particle size distribution, but closer measurement revealed the discontinuity. For this reason it was necessary to obtain spheres from several manufacturers in different lots and to mix them so as to obtain a fairly continuous particle

size distribution. The continuity was checked by withdrawing a sample and applying a condensed version of the calibration procedure to be explained in a later paragraph. The use of a mixture of many different lots of spheres produces a calibration curve with many minor fluctuations.

### 4. Preparation of Samples

A convenient total weight of spheres in the final calibrated sample is about 100 g. The total number of samples prepared in lot 3 was 256 (equal to  $2^8$ ) which required about 56 lb of glass spheres having a continuous range of sizes in the proper proportions. After lot 3 had been assembled and the preliminary check calibration indicated that the particle size distribution would be satisfactory, the individual samples were prepared. The "Boerner Sampler" [6] was used as the sample reduction device.<sup>5</sup> It is a riffle-type apparatus with a funnel, gate, and chute attached above the compartments, which are arranged circularly. The combination of gate and chute makes it impossible to feed too fast or to feed at the wrong angle. It has been found to give at least as good results as any known sample-reducing device or technique. As an additional precaution to neutralize any errors of the sampling device, careful attention was paid to the side the sampler from which the sample emerged. Although 256 samples were desired, 512 samples were first made; these were recombined in pairs so that the final sample represented equal quantities of material from each side of the sampler. Figure 5 illustrates the process for only four samples.

<sup>5</sup> Acknowledgment is gratefully made to Lawrence Zeleny, Grain Branch, Production and Marketing Administration, U. S. Department of Agriculture, for the loan of these samplers.

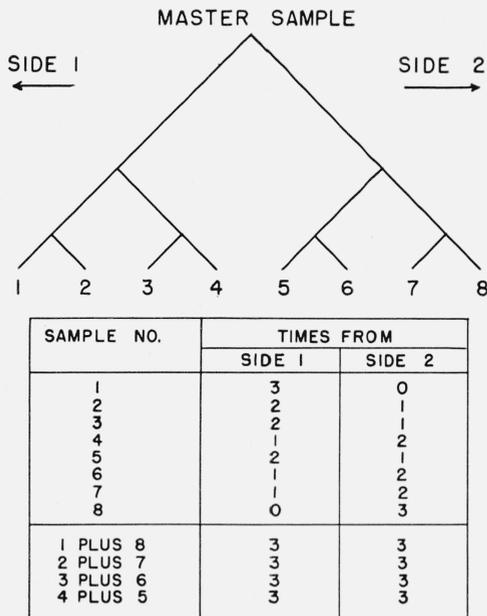


FIGURE 5. Method employed to subdivide mixture of spheres into individual calibration samples.

#### 4.1. Selection of Samples for Calibration

It was recognized that the essential problem in calibration was adequate sampling. The spheres were divided into 256 samples by the best means available, but it must be admitted that all 256 samples may not be identical. Every twenty-fifth sample in the orderly arrangement of numbers given to the samples was chosen as a sample of the 256, making 10 in all. Each of these 10 samples contain approximately 10 million spheres, therefore, they must be further sampled to obtain a practical number small enough to measure. Because spheres of approximately the same size do not tend to segregate and present an easier problem of measurement, each of the 10 samples was separated into 14 sieve fractions.

The spheres for the actual measurement were mounted on a gelatine-coated microscope slide. In order to make sure that an adequate sample was obtained from each sieve fraction, four different slides were prepared, each slide representing particles from a different portion of the sample container. Twenty-five particles chosen at random from each slide were measured. A total of 1,400 particles in each sample was measured; altogether 14,000 particles were measured for the 10 samples.

#### 4.2. Measurement of Diameters

The apparatus for the measurement consisted of a projection microscope with its accompanying light source and screen. The measurements were made on the projection of the image of the particles on the screen. The magnifications and projection distances were arranged so that the projections of the particles were between 6 and 30 cm in diameter. Two

different microscope objectives were used, 16 and 32 mm, according to the magnification desired. The ocular was 7.5 power with a scale mounted inside. The measurements were made in air. A carbon arc and condenser lens system were used for illumination. The magnifications were checked at frequent intervals throughout the measurements by the use of two different stage micrometers. Both of these stage micrometers have been calibrated at this Bureau and the observations obtained are accurate to within 1  $\mu$ .

In order to speed the measurement, a scale was drawn on a separate piece of paper that could be moved about the screen. The distance between the microscope and the image was adjusted so that the graduations on the hand scale exactly coincided with the projected scale in the eye piece. The hand scale could then be moved about, and several beads near the center of the screen could be measured very rapidly. Two people working together measured 25 particles in about 2 minutes. Counting the time required for changing slides, preparing samples, and relaxation to prevent eyestrain, 300 or 400 particles were measured per hour. When nonspherical particles were encountered, the minimum diameter was measured. This was done because it was desired to know the size of the hole through which a particle passes rather than the average size of the particle.

The individual diameters were measured to the nearest 2 to 5 percent, depending upon the part of the scale in use. More precise measurements could have been made. However, since sampling rather than measurement is the factor that limits the accuracy, greater precision was unnecessary and would have greatly lengthened the subsequent work of computation. As it was, 88 points were obtained in the range 60 to 1200  $\mu$ , averaging about five points within the range between each pair of sieves of the  $4\sqrt{2}$  series.

### 5. Computation

The measurements of the diameter of each sphere were recorded in terms of the projected scale reading. Since it was desired to calculate the weight fraction finer than each size, the first step was to determine the diameter frequency distribution within the 100 spheres representing one sieve fraction of one sample. The actual diameter for each size was evaluated by multiplying the scale reading by the magnification factor. These computations give a number-size distribution. On the assumption that all the particles were of the same density and same shape, the weight is proportional to the number of particles and to their diameter cubed. The weight fraction of spheres of diameter,  $d$ , in the sieve fraction thus becomes  $nd^3/\Sigma nd^3$ . If  $w$  is the weight of the sieve fraction and  $\Sigma w$  the total weight of the sample, then  $w/\Sigma w$  is the weight fraction of the sieve fraction in the total sample. The weight fraction ( $f$ ) of spheres of diameter  $d$  in the total sample is

$$f = \frac{nd^3}{\Sigma nd^3} \times \frac{w}{\Sigma w}$$

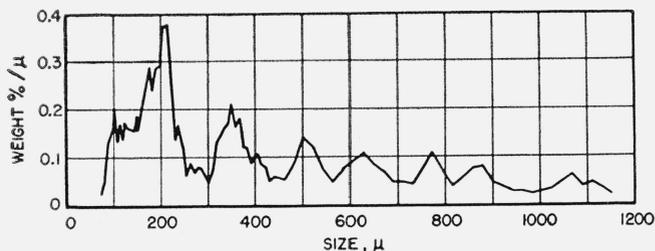


FIGURE 6. Differential particle-size distribution curve of mixture of spheres.

Calibrated glass spheres, lot 3.

### 5.1. Sample Computation

Typical data are given in table 1 for one sieve fraction of one sample. Each diameter recorded in table 1 is the mean of the size range; for example, measurements between 1.875 and 1.925 are recorded as 1.90. The frequency distribution, diameters, and the computations leading to the weight fractions are given in table 2.

The differential type of particle size distribution (expressed by weight fraction per unit size range as a function of size) is obtained by dividing each  $f$  by its corresponding size range. The integral type of particle size distribution (expressed by weight fraction finer as a function of size) is obtained by summing the  $f$ 's. The particle size associated with the sum of the fractions finer is the smaller limiting diameter of the size range. This is in contrast to the mean of the size range used in the above expression for  $f$  and in the differential type of particle size distribution.

These computations were repeated for each fraction of the ten samples, and the values were averaged to obtain the final calibration. The calibration is given graphically in figures 6 and 7.

### 5.2. Evaluation of Sieve Opening

In order to evaluate the sieve opening with a sample of the spheres, the entire sample is placed on the sieve or sieves in question. The sieves are shaken with the calibrated spheres in the same manner as will be used with the unknown material. The spheres are then carefully brushed from each sieve and the weight fraction finer than each sieve evaluated. The effective size of the sieve is then read directly from the calibration curve (fig. 7). Care must be exercised to avoid loss of the spheres in order that the sample can be used again. Experience has shown that the fines are most easily lost; therefore, if the total weight of the sample decreases slightly with repeated use, it may be assumed that the loss is in the finest sizes. The particular operation in which there is most chance of losing particles is the transfer from the sieve to the weighing container. The loss of particles in this operation can be minimized by inverting the sieve into a deep funnel and removing the contents with a stiff brush. The stem of the funnel should fit snugly into the receiver.

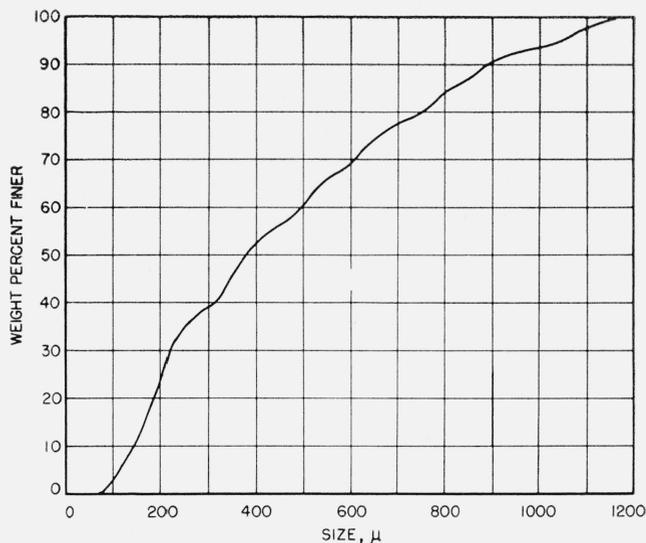


FIGURE 7. Integral particle-size distribution curve of mixture of spheres.

Calibrated glass spheres, lot 3.

TABLE 1. Typical data obtained for observations of particle diameters

Measured diameters of individual particles on microscope slide—			
1	2	3	4
2.00	1.80	1.95	1.90
2.05	1.95	1.85	1.70
1.80	1.85	2.10	1.80
1.80	1.85	1.85	1.75
1.80	1.85	1.75	1.80
1.90	1.80	1.75	1.80
1.95	1.80	1.90	1.85
1.75	1.80	1.80	1.80
1.90	1.90	1.90	1.80
1.85	1.80	1.95	1.85
2.05	1.80	2.00	1.90
1.90	1.85	1.80	1.80
2.05	1.90	1.90	1.95
1.80	1.85	1.65	2.00
1.75	1.80	1.90	1.80
2.00	2.00	1.75	1.75
2.00	1.80	1.95	1.70
1.80	1.90	1.75	1.85
1.85	1.85	1.80	1.80
2.00	1.85	1.85	1.95
1.90	1.90	1.95	2.05
1.80	1.80	1.85	1.95
1.85	1.90	1.80	2.00
1.85	1.85	1.85	1.75
1.80	1.90	1.90	1.85

Identification number of sample: 100  
 Sieve fraction: through 20 on 25  
 Weight of fraction:  $w=8.78$  g  
 Total weight of sample:  $\Sigma w=106.51$  g  
 Magnification factor: 1 unit=419  $\mu$

### 5.3. Application to Sieve Analysis

One of the most disturbing features of sieve analyses is the inability to obtain the same results with the same sample using different sets of sieves that have been considered identical. The small variations in the sieve openings inherent in the manufacture of the sieves are the cause of this trouble. In order to

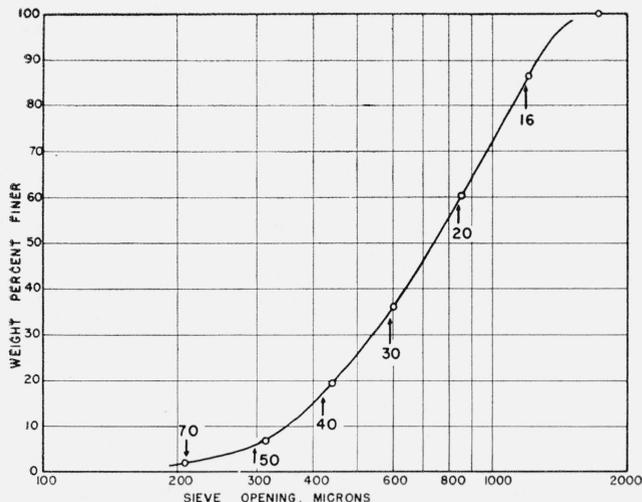


FIGURE 8. Corrections of sieve analysis to nominal sieve opening, bone char 32; arrow indicates nominal sieve opening.

TABLE 2. Computation of weight fraction from measured diameters of 100 particles

Measured diameter, scale units	Diameter, $d$	Number, $n$	$d^3$	$nd^3$	$f$
2.10	879.9	1	$6812 \times 10^5$	$6812 \times 10^5$	0.0011
2.05	859.9	4	6337	25348	.0040
2.00	838.0	8	5885	47080	.0074
1.95	817.1	9	5454	96166	.0151
1.90	796.1	17	5045	85765	.0135
1.85	775.2	20	4658	93160	.0146
1.80	754.2	29	4290	124410	.0195
1.75	733.3	9	3942	35478	.0056
1.70	712.3	2	3614	7228	.0011
1.65	691.4	1	3304	3304	.0005
		$\Sigma = 100$		$\Sigma = 524751$	$\Sigma = .0824$

$$\frac{w}{\Sigma w} = \frac{8.78}{106.51} = 0.0824 \text{ (see table 1)}$$

$$f = \frac{nd^3}{\Sigma nd^3} \times \frac{w}{\Sigma w} = \frac{nd^3}{524,751 \times 10^5} \times 0.0824 = 1.5709 \times 10^{-12} nd^3$$

reduce the results of any sieve analysis to that which would be obtained if sieves with nominal sized openings were used, it is only necessary to plot the results as cumulative percentage finer (or coarser) as a function of the calibrated opening of the sieve. Such a plot for a bone char (char 32) is shown in figure 8. From this curve the cumulative percentage finer (or coarser) than the nominal openings can be obtained (at points indicated by arrows); hence the corrected sieve analysis may be calculated. In this way sieve analyses made at different laboratories with different sets of sieves may be reported with reference to the same sieve openings.

## 6. Evaluation of Errors

The errors inherent in the method of measuring the effective size of the openings of testing sieves by means of calibrated glass spheres arise from three sources. First, a *dividing error* occurs when the samples are prepared. Second, a *sieving error* occurs

when the samples are used. Third, a *calibration error* occurs in the sampling and measuring involved in the calibration process. The magnitude of each of these errors has been determined and, also, their effect on the over-all reproducibility and accuracy of the method.

The dividing error can be considered in two parts. One is due to the inaccuracies of the sample-reducer, and the other arises from the use of only a relatively limited number of particles. It is possible to estimate the error of the latter by the application of statistics. If  $n$  is the number of particles of one size remaining in the final sample after three or more separations on the sample divider, then according to statistical theory (appendix 1), the standard deviation associated with this number is approximately  $\sqrt{n}$ . The error in the determination of size of particle from this cause can be readily evaluated (appendix 2) and is listed in table 3.

TABLE 3. Errors in the measurement of effective openings of testing sieves by means of glass spheres

U. S. Standard Sieve No.	Standard deviation		
	Dividing	Sieving	Calibration
	<i>Microns</i>	<i>Microns</i>	<i>Microns</i>
18	3.56	2.6	2.8
20	1.40	3.5	2.0
25	1.42	2.3	1.5
30	0.74	1.2	1.24
35	.30	2.4	1.14
40	.46	2.0	1.07
45	.15	3.1	0.98
50	.23	0.7	.88
60	.10	1.6	.79
70	.04	1.2	.69
80	.04	0.8	.66
100	.03	.3	.63

Because of the good construction of the sampling device and the additional precautions that were observed, it is reasonable to assume that the error arising from the inaccuracies of the sampling device is small. Indeed, it can be shown that this error is so small that it exerts no appreciable influence on the final sum of all errors (appendix 3).

The error due to sieving depends upon the particular sieving method employed. In order to obtain some idea of the error that might be encountered, one sample was sieved nine times, using the particular procedure used in this laboratory.<sup>6</sup> The standard deviations representing the variations among these nine sievings are also listed in table 3. These variations are due to sieving alone because the same sample, the same sieves, and the same calibration curve were used.

The variation among the calibrations of the ten different samples is a measure of the sum of the dividing and calibration errors. The dividing error has already been determined and can be subtracted<sup>7</sup> from the sum to yield the calibration error alone (appendix 4). This error is also listed in table 3.

<sup>6</sup> The sieves were shaken for 8 min in an old style (two eccentric) Ro-Tap operating at 117 taps/min.

<sup>7</sup> The variance (standard deviation squared) is additive.

It is noted that each of the errors are of the same order of magnitude, indicating that any additional precautions in the sample dividing or calibration would have been wasted effort unless some method of sieving with less error could have been found.

The accuracy and reproducibility of the glass sphere method of calibrating testing sieves are tabulated in table 4. The values listed are the maximum variations that might reasonably be expected and are taken as three times the standard deviations. The accuracy is measured by the sum<sup>7</sup> of the calibration and dividing errors and is an indication of the precision with which the size of the spheres is known.

There are two types of reproducibility. The variation to be expected when the same sieve is calibrated several times with the same sample of glass spheres is that due to the sieving error. If different samples of glass spheres are used, then the dividing error is included also.

As a general summary in regard to errors it can be stated that both the accuracy and reproducibility of a glass sphere calibration is about 1 percent of the size of the sieve opening. This is significantly better than the 2- to 7-percent variation allowed in the present specifications for testing sieves. Even more important, however, the glass spheres measure the effective opening (for particles not too far from spherical) rather than the average opening.

TABLE 4. *Reproducibility and accuracy of the calibration process*

The values listed are the maximum variation to be expected (three times the standard deviation)

U. S. Standard Sieve No.	Nominal opening	Reproducibility		Accuracy
		Using same calibration sample	Using different samples	
	<i>Microns</i>	<i>Microns</i>	<i>Microns</i>	<i>Microns</i>
18	1,000	7.8	13.3	13.5
20	840	10.5	11.4	7.5
25	710	6.9	8.1	6.3
30	590	3.6	4.2	4.4
35	500	7.2	7.3	3.5
40	420	6.0	6.1	3.4
45	350	9.1	9.1	2.9
50	297	2.1	2.2	2.8
60	250	4.8	4.8	2.4
70	210	3.7	3.7	2.1
80	177	2.4	2.4	2.0
100	149	0.9	0.9	1.9

## 7. References

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## 8. Appendix 1

Acknowledgment is made to J. M. Cameron and John Mandel of the Bureau for the derivation leading to the following results.

A large number of particles is split into two groups as evenly as possible. Each of these groups is again divided into two groups as evenly as possible, and so on. After  $k$  such splitting operations,  $2^k$  subgroups are formed.

If there were  $N$  particles in the original aggregate, one would expect each subgroup to have  $N/2^k = n$  particles. But, due to randomness in the partitioning, the actual number of particles observed will vary about this expected number. As few as zero could be found or as many as  $N$ .

Assuming that each particle has an equal chance of going into either group at each partition, the probability that a particle be found in a particular subgroup is  $1/2^k$ , and the numbers of particles in the subgroups will be binomially distributed. From the mathematics of the binomial distribution, the standard deviation of the number of particles found in the various subgroups is

$$\sigma = \sqrt{N \frac{1}{2^k} \left(1 - \frac{1}{2^k}\right)} = \sqrt{n \left(1 - \frac{1}{2^k}\right)}$$

It may be noted that for values of  $k$  greater than 3 the value of the factor in parenthesis is practically one, hence, the standard deviation becomes

$$\sigma = \sqrt{n} \quad (\text{for } k > 3).$$

## 9. Appendix 2

In weight units the standard deviation is  $\sigma_w = \sqrt{n \rho s d^3}$ , where  $\rho$  is the particle density (2.6 g/cm<sup>3</sup> for glass),  $s$  is the shape factor ( $s = \pi/6$  for spheres), and  $d$  is the particle diameter. If  $w$  is the weight of a fraction of particles all of the same size then the number of particles is obtained by

$$n = \frac{w}{\rho s d^3}$$

The standard deviation in weight units thus becomes

$$\sigma_w = \sqrt{\frac{w}{\rho s d^3} \rho s d^3} = \sqrt{w \rho s d^3}$$

The weight fraction is obtained by dividing by the total weight of all the fractions.

$$\sigma_f = \frac{\sigma_w}{\sum w} = \sqrt{\frac{w \rho s d^3}{\sum w}}$$

Since the error is small, to a first approximation the ratio of error in weight fraction units to the error in size units is equal to the slope of the calibration curve at that point. That is,  $\sigma_f/\sigma_s = df/ds$  where  $df/ds$  is the slope of integral type particle size distribution curve (fig. 7) and is equal to the differential particle size distribution given in figure 6. The standard deviation in size units can thus be expressed in terms of easily measurable quantities as

$$\sigma_s = \sqrt{\frac{w \rho s d^3}{\sum w} \frac{df}{ds}}$$

This quantity is tabulated in table 3 for each of the sieve fractions.

## 10. Appendix 3.

In the course of the calibration procedure the 10 samples were all sieved. Nine of these samples were sieved with the same set of sieves and in the same manner. The variance among these sieve analyses is listed in table 5, column 5, and is the sum of dividing and sieving errors. No calibration error is involved because all sievings are referred to the same calibration curve. According to statistical theory, when errors are independent, their variances (standard deviation squared) are additive. In table 5 are listed the variances due to dividing error (col. 2), sieving error (col. 3), and their sum (col. 4). The sums are compared to the measured combined errors from the nine different samples (col. 5) by taking the ratios, and applying the *F* test, (col. 6). In no case does the value of *F* indicate a highly significant difference between the two. On one-half the sieves column 4 is larger, and on the other half column 5 is larger. Altogether, this indicates that there is really no difference between the two columns.

TABLE 5. Variance due to dividing and sieving

U. S. Standard Sieve No.	Dividing	Sieving	Dividing +sieving calculated	Dividing +sieving observed	F <sup>a</sup> Col. 5 Col. 4
1	2	3	4	5	6
	$\mu^2$	$\mu^2$	$\mu^2$	$\mu^2$	
18	12.7	6.9	19.6	30.8	1.57
20	1.96	12.5	14.5	16.0	1.11
25	2.02	5.3	7.3	10.2	1.40
30	0.55	1.4	2.0	7.8	3.94
35	.09	5.9	6.0	3.6	0.60
40	.21	3.9	4.1	2.9	.70
45	.02	9.3	9.3	2.3	.24
50	.05	0.51	0.56	1.96	3.50
60	.01	2.50	2.51	1.00	0.40
70	0	1.53	1.53	0.49	.32
80	0	0.62	0.62	.25	.40
100	0	.09	.09	.16	1.78

<sup>a</sup> For 8 and 8 degrees of freedom, *F* at 1-percent level = 6.03, *F* at 5-percent level = 3.44.

Column 5 includes errors due to inaccuracies in the sample divider, while column 4 does not. This indicates, therefore, that the error due to the sample divider is so small as to be negligible.

## 11. Appendix 4

The variance among the calibrations of the 10 different samples is a measure of the sum of the dividing and calibrating errors. It is listed in column 2 of table 6 and is composed of two parts, one due to dividing the samples (col. 3) and the other due to the calibration procedure. A subtraction yields the variance due to calibration alone (col. 4). This is the variance among the 10 samples. The variance of the mean from the true value is one-tenth of the variation among the 10 samples and is listed in column 5. The sum of the dividing variance and calibration variance of the mean is listed in column 6. This is a measure of the precision and in this case, considering the whole procedure, it is a measure of the accuracy.

TABLE 6. Variance due to dividing and calibration errors

U. S. Standard Sieve No.	Dividing+ calibration measured	Dividing	Calibration	Calibration variance of the mean	Dividing+ calibration variance of the mean
1	2	3	4	5	6
	$\mu^2$	$\mu^2$	$\mu^2$	$\mu^2$	$\mu^2$
18	90.4	12.7	77.7	7.77	20.5
20	43.6	2.0	41.6	4.16	6.2
25	25.0	2.0	23.0	2.30	4.3
30	16.0	0.6	15.4	1.54	2.1
35	13.0	.1	12.9	1.29	1.39
40	11.6	.2	11.4	1.14	1.34
45	9.6	.0	9.6	0.96	1.96
50	7.8	.1	7.7	.77	0.87
60	6.3	0	6.3	.63	.63
70	4.8	0	4.8	.48	.48
80	4.4	0	4.4	.44	.44
100	4.9	0	4.0	.40	.40

WASHINGTON, November 24, 1950.