

The two values for the heat of dissociation differ by the rather large amount of 626 j. Cottrell and Wolfenden state that their result is subject to considerable uncertainty, for it represents a difference between two experimental quantities and also rests upon a rather arbitrary extrapolation. Differences of extrapolation often do not affect the temperature coefficient, and it is perhaps significant that practically identical values of ΔC_p° , that is $d(\Delta H^\circ)/dT$, were obtained by the two methods. Cottrell and Wolfenden found ΔH° to pass through zero at 26° C, in reasonably good agreement with 23° C at which the value of $-\log K_2$ given in table 3 reaches a minimum.

V. References

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Methods of Sieve Analysis With Particular Reference to Bone Char¹

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The procedure for separating particle sizes of solid adsorbents by sieving has been studied in detail because of the influence of the particle size on adsorbent properties. The investigation was conducted chiefly with sieve openings in the range between U. S. Standard Sieves No. 8 and No. 80. The largest source of error is in the testing sieves themselves. This is due to the tolerances permitted by the present specifications. It is feasible to calibrate testing sieves by the use of a calibrated sample of spherical glass beads and thus obtain the opening that is effective in sieving. The calibration of testing sieves in this manner can lead to reproducible sieve analyses by different laboratories. A simple procedure is proposed to determine the uniformity of sieve openings and, thereby, to furnish a criterion for the discard of distorted sieves. An analysis with seven Ro-Tap machines indicated that, in general, best results are obtained when the Ro-Tap is operated at 115 taps/min of the knocker mechanism. The other variables concerned with shaking that were examined are of minor importance and need not be rigidly controlled. As first choice, the weight of the sample should be between 100 and 150 g. The shaking time should be adjusted to the weight and the particle size distribution of the sample according to relationships developed.

I. Introduction

The adsorbent properties of bone char and other materials are greatly influenced by particle size.

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Sieve analysis is one of the basic tests for measuring the particle size of all powdered and granular materials. In sugar refining and many other industries great importance is ascribed to sieve analyses in spite of the fact that their reproducibility may be extremely poor, in comparison with other measurements such as volume, mass, or chemical composition. Differences in the results of sieve analyses of

the order of magnitude of 10 percent are frequently reported between different laboratories, while differences of 1 or 2 percent are not uncommon with the same operator and the same sieving equipment. In view of the importance of sieve analysis and of the poor reproducibility when compared to other measurements, an investigation was made of the sources of errors and of the steps that might be taken to reduce the errors to a minimum. Previous work on this subject is not considered adequate [1, 2].³

The sources of errors in a sieve analysis can be classified under three general headings: The sieves, the method of shaking, and the sample. The sieves considered are the standard 8-in. laboratory testing sieves. The nominal sizes of the openings of the standard sieves ($\sqrt{2}$ series) are in a geometric series with a fixed ratio of the square root of 2. For closer sizing, an additional sieve is provided between each pair in the standard series thus forming the closest-sizing series ($\sqrt[4]{2}$ series). The methods of shaking the sieves that were studied were by hand and by the use of the Ro-Tap⁴ machine. Although there are other shaking devices, the Ro-Tap machine, which is one of those commonly employed, was the only type studied. The majority of the samples studied were bone chars from various cane sugar refineries throughout the world. Other materials included were crystalline refined sugar, glass, sand, iron filings, and granular bismuth. The particle shapes included were spherical, irregular, and long needle-like granules. The particle sizes ranged from those passing a No. 4 sieve⁵ to those retained on a No. 270 sieve. These correspond to particle diameters of 0.476 to 0.0053 cm, or 0.187 to 0.0021 in. Most of the work was conducted in the sieve size range No. 8 to No. 80.

The application of the results of a sieve analysis to the interpretation of physical properties presents many difficult problems, and these are not considered here in all their aspects. DalleValle [3] has considered many phases of sieve analysis, but unfortunately these were not all examined critically by him. Hatch [4] considered the relations between weight-size and number-size distribution and also the various methods of evaluating the average particle size. The relation between the size of the sieve openings and the average diameter of irregularly shaped particles that will pass through them has not been adequately investigated. The method of attack emphasized in this investigation makes use of glass spheres to define the effective openings of sieves.

1. Method of Reporting Sieve Analyses

Sieve analyses are usually reported as percentage by weight of the total sample that passes a certain sieve and that is retained on another, for example, "percentage passing No. 20 and retained on No. 30". This is frequently shortened for convenience to, for example, "20 to 30 fraction" or "20 to 30 mesh". The use of the term *mesh* in this sense is to be avoided

[5]. For some comparisons, sieve analyses are reported as cumulative percentage finer or coarser than a certain sieve.

When evaluating differences between sieve analyses, the differences are expressed in percentage of the total sample for a certain sieve fraction. If the difference (or deviation) is compared to the mean value, then a percentage of the percentage of the total sample is obtained. Such a terminology is awkward, and there is used instead the phrase "coefficient of variation", which is expressed in percentage.

In evaluating the variation among several sieve analyses, the standard deviation is computed for each sieve in the usual manner. The standard deviations for the different sieve fractions are not strictly comparable for reasons that will be apparent, although they have been averaged in some cases to give a single figure as a measure of the reproducibility of the sieve analyses.

II. Sieves

One of the largest sources of variation in sieve analyses is in the testing sieves themselves. The wire size, average opening, and uniformity of opening are specified⁶ with a small variation allowed in each for manufacturing tolerances. The wire size is of minor consequence, because it does not directly influence the size of particle that will be retained on the sieve.

1. Variation in Average Opening

The variation in average opening allowed by the specifications may seem stringent from a manufacturing point of view; but it has an important effect on sieve analyses, since the variation ranges from ± 3 percent for sieves No. 16 and coarser to ± 7 percent for sieves No. 200 and finer. For example, the variation allowed for sieves No. 20 and 30 ($\sqrt{2}$ series) is ± 5 percent of the nominal opening. The No. 20 sieve, whose nominal opening is 0.084 cm, thus may have average openings ranging between 0.080 and 0.088 cm. Similarly, No. 30 sieves of nominal opening 0.059 cm may have average openings somewhere between 0.056 and 0.062 cm. When these two sieves, both uniformly woven, are used to define a sieve fraction, the particle sizes that are retained between the two sieves depend on the openings in both sieves. The mean value of the openings can, therefore, indicate the probable spread in particle sizes. The maximum spread using these two sieves is 0.032 cm, and the minimum is 0.018 cm. It is noted that the maximum would be *twice* that of the minimum.

This type of error is much larger when sieves of the $\sqrt[4]{2}$ series are used. This is best explained on a percentage basis. The nominal opening of each sieve is only 19 percent larger than that of the next smaller. When sieves No. 200 or finer are used, and the average opening of the larger sieve is oversize to

³ Figures in brackets indicate the literature references at the end of this paper.

⁴ Manufactured by W. S. Tyler Co., Cleveland, Ohio.

⁵ All sieve numbers, unless otherwise noted, refer to the U. S. Standard series.

⁶ Identical specifications are issued by the American Society for Testing Materials (ASTM) [6], The American Standards Association (ASA) [7], and the National Bureau of Standards [5].

the limit of the specifications, and the average opening of the smaller sieve is undersize to the limit of the specifications, then this 19 percent becomes approximately $19+7+7=33$ percent. In the converse case, the figure becomes $19-7-7=5$ percent. If the size distribution by weight of the material being sieved is approximately constant, then the amount of material remaining between the two sieves in the first case would be nearly *seven* times as much by weight as in the second case, the sieves being uniformly woven. Sieves that did not conform to specifications might give variations many times greater than this. With careful selection sieves can be held to tolerances much closer than the specifications, with proportionally better results. For the best results it would be necessary to calibrate individually each sieve to find the actual effective openings of that particular sieve. When reproducibility alone is desired and accuracy of measurement of particle size of minor importance, sieves may be carefully matched.

2. Calibration of Sieves

Testing sieves are calibrated from measurements with a projection microscope (see [5]). For a small fee, the National Bureau of Standards calibrates sieves by this method. This service is generally available only for new sieves. In a calibration of this type, the thickness of the wires is measured, and the number of openings per inch is counted usually for a distance of at least 6 in. The average opening is then determined by dividing the difference between the total distance measured and the sum of all wire diameters contained in this distance by the number of openings.

It is to be noted that if the size of the openings is not quite uniform, then the larger openings are the effective ones, and the average opening is almost meaningless. Weber and Moran [8] suggested an absolute microscopic calibration of testing sieves consisting in measuring a large number of the openings and determining the effective size of the openings by use of an empirical relation between the statistical parameters and the effective opening. This calibration method may be too long and involved to be of practical value. However, when particles to be sieved are fairly symmetrical in shape, the effective opening of a sieve can be easily determined by measuring the size of spherical particles that will pass. This type of measurement is best done by means of calibrated samples of material of known particle-size distribution, determined by some means other than sieving. This method has the advantage of being quickly done without special equipment by anyone familiar with sieving procedures. It also has the advantage that used sieves may be checked periodically to determine whether wear or deformation has occurred to an extent sufficient to make them unsuitable.

The material of the calibrated sample should be hard enough to eliminate completely any question of abrasion. The best shape of the particles for calibration purposes is spherical, because the

diameter of the particles can be measured by microscopic means, and there is no doubt about the orientation of the particle as it passed the sieve opening. Glass beads of the type used for highway markings are suitable for this use when properly selected.⁷

3. Glass Beads as Reference Samples for Calibration of Sieves

Spherical glass beads ranging in size from those passing a No. 20 sieve to those retained on a No. 100 sieve were used to explore the possibilities of calibrating testing sieves. With this sample, sieves from No. 25 through No. 80 can be calibrated. A sorting procedure was developed to remove all misshapen particles from the material as received. The material was then divided into smaller quantities by a sample divider of the riffle type. One of these samples was sieved into 10 fractions so beads of nearly the same size would be grouped together for ease of measurement. Four samples of about 100 beads were taken from each of these 10 fractions and a microscope slide prepared of each. Twenty-five beads from each slide were measured in air with a micrometer microscope.

From the measured diameters of 1,000 particles, the particle-size distribution was evaluated. At least 10,000 particles are desirable for accurate results. However, as the object of this experiment was only to explore the feasibility of such a calibration sample, this refinement was not considered necessary. Figure 1 is a plot of the particle-size distribution for the glass beads. The percentage by weight finer than any specific size is plotted against that size of bead as determined microscopically.

4. Procedure in Using the Glass Beads

In order to calibrate any one sieve, it is merely necessary to place the entire sample on the sieve, shake until the rate of passage of beads through the sieve is practically zero,⁸ and then carefully weigh the beads that have passed the sieve and calculate the percentage by weight. The effective opening of the sieve is then read directly from the calibration curve. When several sieves are to be calibrated at the same time, they can be nested and shaken together. The weight finer than any particular sieve is the sum of the weight on all the sieves below and in the pan.

A numerical example of the procedure may be instructive. A single sieve (No. 45) was tested with a glass-bead mixture weighing 110.06 g. It was found that 25.96 g passed the sieve. Hence, the percentage by weight passing was $(25.96/110.06) \times 100 = 23.6$ percent. From the calibration curve for this sample (see fig. 1) the effective sieve opening is read as 372μ . The nominal opening of a No. 45 sieve is 350μ , with an allowable variation of ± 5 percent. The effective opening of this sieve is 6.3 percent larger than the nominal size. As will be explained later, such an

⁷ Suitable beads have been obtained from the Cataphote Corp., Toledo, Ohio; Potter Bros. Inc. Ozone Park, N. Y.; Minnesota Mining & Manufacturing Co., Saint Paul, Minn.

⁸ For details see section III, 2.

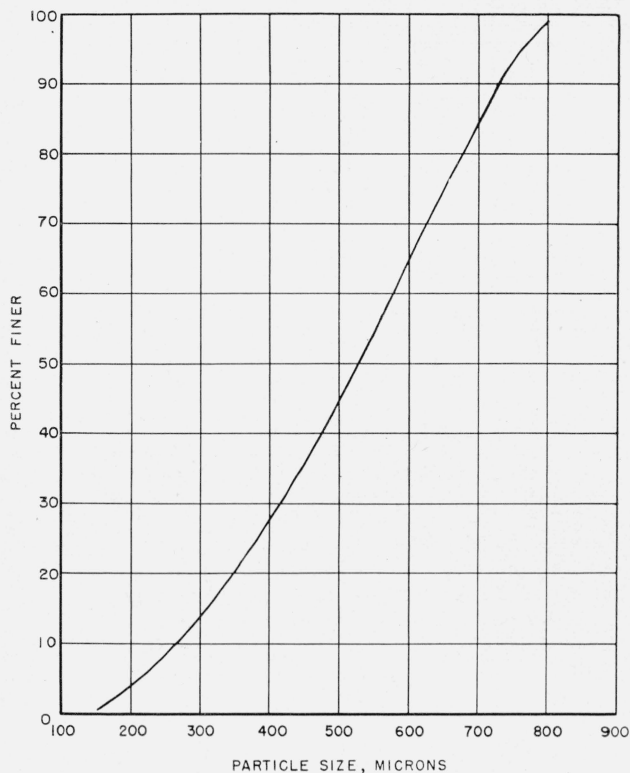


FIGURE 1. Particle size distribution of glass beads for calibration of testing sieves.

effect might reasonably be expected; in using this sieve for testing purposes, it is desirable to take into account the variation of its effective opening from the nominal opening.

5. Application of Correct Sieve Openings to Sieve Analyses

One of the most disturbing features of sieve analyses is the inability to obtain the same results with the same sample when using different sets of sieves. It has been pointed out that the small differences in the sieves inherent in manufacturing is the cause of this trouble, and that with suitable calibration corrections can be made for these differences. Also, if sieves that are not of standard sizes are used, the results of the sieve analysis can be reduced to what would be obtained if nominal-size sieves were used.

A simple procedure is to plot the results of sieve analysis as cumulative percentage finer as a function of the effective opening of the sieve. From this curve the cumulative percentage finer that would pass through the nominal openings can be obtained and, hence, the corrected sieve analysis may be calculated. An example of this procedure will now be given. A sample of service bone char was sieved with two different sets of sieves. Both were good sieves and supposed to be equivalent. The results are contained in table 1.

Both of these sets of sieves were then calibrated by use of the glass beads with the results given in table 2. It is to be noted that in every case the calibrated

TABLE 1. Sieve analysis of a service bone char with two different sets of sieves

U. S. Standard Sieve No.	Char retained on—		Ratio of percentage retained on set 1 to that on set 2
	Set 1	Set 2	
25	Percent 19.3	Percent 17.4	1.11
30	5.2	7.6	0.68
35	7.2	7.2	1.00
40	10.5	7.0	1.50
45	11.3	12.6	0.90
50	15.7	13.7	1.15
60	14.0	18.0	0.78
70	10.9	8.8	1.24
80	2.5	4.8	0.52
Through 80	3.4	2.9	1.17

opening is larger than the nominal opening. This is because this method of calibration measures an effective opening that is larger than the arithmetic average of the projections of all the openings. Moreover, 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 always larger than its projection on the plane of the sieve cloth. The sieves were manufactured to have the average opening equal to the nominal opening within the specified tolerances.

The two sieve analyses are plotted as a function of their effective openings in figure 2. The two curves very nearly coincide. From these curves the

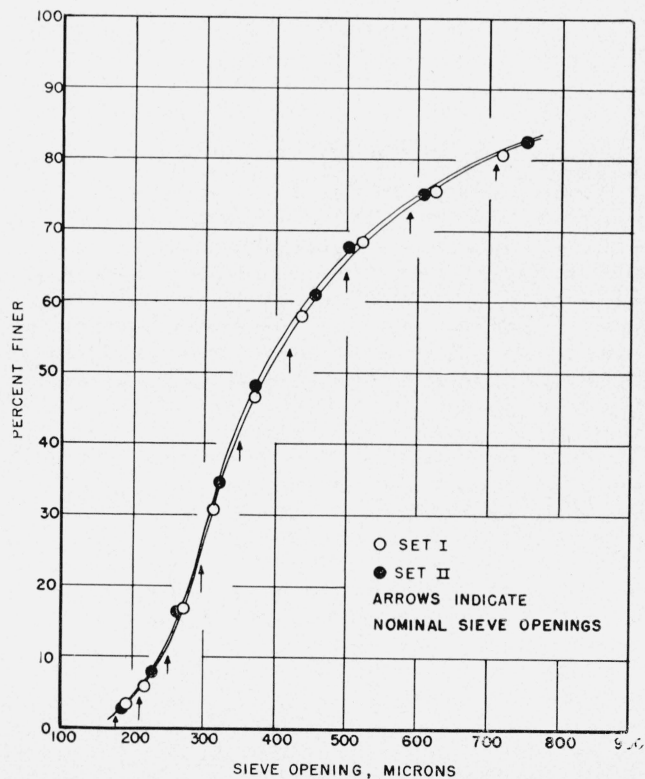


FIGURE 2. Sieve analyses of the same sample with two sets of sieves.

TABLE 2. *Effective openings of two sets of sieves*
[1,000 μ equal 1 mm.]

U. S. Standard Sieve No.	Effective openings		Nominal opening
	Set 1	Set 2	
25	719	752	710
30	627	610	590
35	523	503	500
40	436	456	420
45	372	374	350
50	314	322	297
60	272	264	250
70	217	228	210
80	193	186	177

corrected analyses are read at the nominal openings (indicated by arrows). The corrected analyses are tabulated in table 3. The excellent agreement of the corrected sieve analyses indicates the value of the calibration method.

TABLE 3. *Corrected sieve analyses with different sets of sieves*

U. S. Standard Sieve No.	Corrected amount retained on—		Ratio of percentage retained on set 1 to that on set 2
	Set 1	Set 2	
	<i>Percent</i>	<i>Percent</i>	
25	19.7	19.4	1.02
30	7.2	7.0	1.03
35	7.1	6.8	1.04
40	10.6	10.4	1.02
45	14.0	14.1	0.99
50	16.5	15.9	1.04
60	12.6	14.0	0.90
70	7.0	7.1	0.99
80	3.4	3.2	1.06
Through 80	1.9	2.1	1.10

6. Uniformity of Openings

Besides the average size of the openings, the uniformity of openings plays an important role in sieve analysis. Before sieves are calibrated for the average effective opening they should be checked for uniformity. In figures 3 and 4 are seen two No. 10 sieves. At first glance the two look alike, but the wires in figure 3 are a little thinner than in figure 4. Both wire diameters are within specifications and, as previously stated, this difference is of minor importance. More careful examination of figure 3 reveals that the wires are not perfectly parallel, and that there is considerable variation in the size of the openings. In figure 4 the variation in the openings is scarcely discernible without the aid of measuring instruments. The sieve of figure 3 has been used for 19 years and has been subjected to rough treatment. The sieve shown in figure 4 has been used very little and still conforms to specifications.

If there are many oversized openings, as in figure 3, then many particles that should remain on the sieve pass through these openings, and the effective size of the sieve is not its average opening but more nearly its maximum opening. If only a few of the openings are very much oversize, material continues to pass these few maximum openings for a long time.

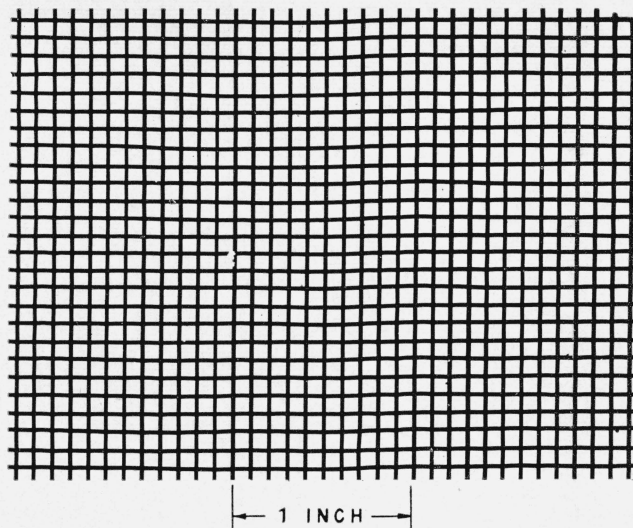


FIGURE 3. *U. S. Standard No. 10 Sieve that is visibly deformed.*

The rate of passage of material through a uniform sieve drops to a very low value after a few minutes of shaking, whereas material continues almost indefinitely to pass the few oversize openings of a non-uniform sieve.

The rate of passage of certain materials through a sieve can be taken as a measure of uniformity of the sieve. In figure 5 are seen sieving curves⁹ for five different sieves. All sieves were nominally No. 40, and the same sample of glass beads was used for each. It must be remembered that it is the sieving rate (slope) rather than the actual weight passing that is of primary interest. Curve A of figure 5 was obtained from a badly distorted sieve whose meshes were in a condition similar to those shown in figure 3. Curve B was obtained from a badly worn sieve that was no longer used for testing. Curve C was obtained from a sieve that had been in

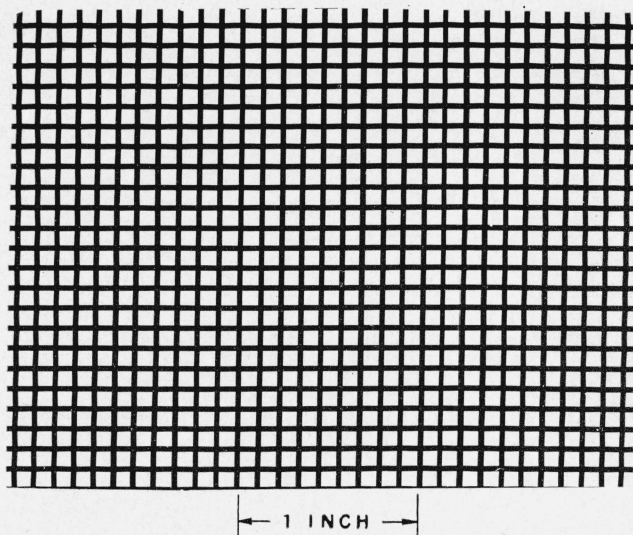


FIGURE 4. *U. S. Standard No. 10 Sieve that is in excellent condition.*

⁹ Explained in detail in section III, 1.

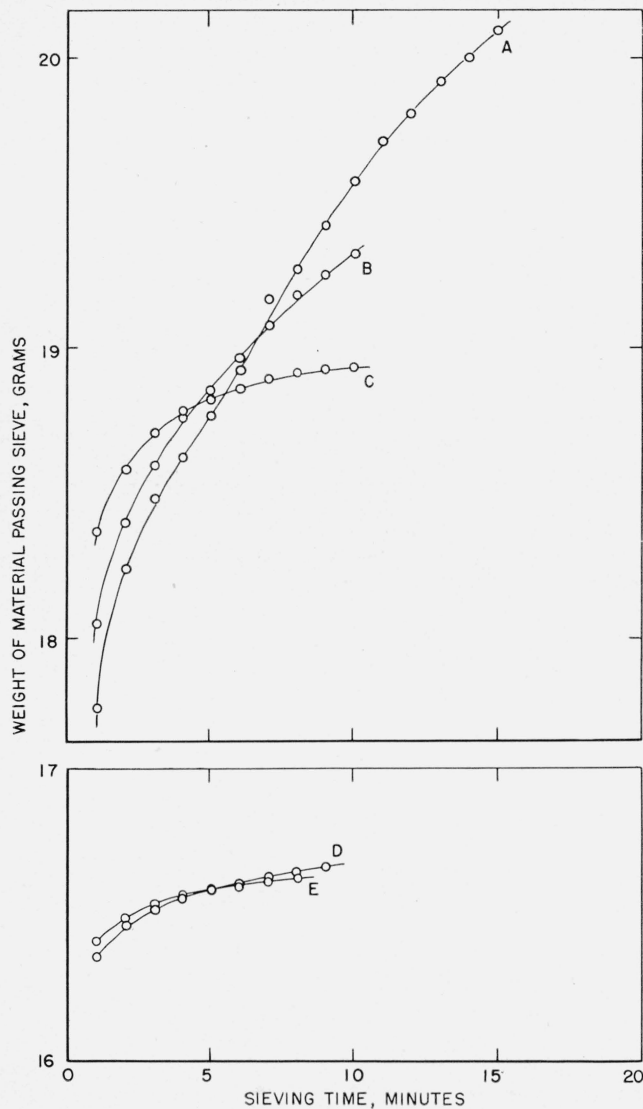


FIGURE 5. Sieving curves (weight passing as a function of time) for sieves of varying uniformity with the same sample of glass beads.

All sieves were No. 40.

use for some years. Curves *D* and *E* were obtained from new sieves that were certified by this Bureau to be within one-half the uniformity tolerances allowed in specifications for testing sieves. The sieves corresponding to curves *C*, *D*, and *E* are considered sufficiently uniform to be of value as testing sieves, whereas those corresponding to curves *A* and *B* cannot be expected to give accurate analyses.

III. Method of Shaking Testing Sieves

The shaking methods considered were by hand and by use of the Ro-Tap machine. The standard sieving methods of the American Society for Testing Materials (ASTM) for roofing materials [9, 10] and plastic molding powders [11] prescribe the machine method as standard. The ASTM methods for testing cement

[12], soap [13], powdered coal [14], refractories [15], road materials [16], and fine and coarse aggregates used in concrete [17] all prescribe hand sieving as standard, but machine sieving is acceptable for all except cement [12], provided the results agree with hand sieving. In hand sieving, the sieves are shaken one at a time until the rate of passage of material through the sieve decreases to some very low value¹⁰ prescribed by the specifications for the particular test. Hand sieving is time consuming and tedious and, moreover, only one sieve is shaken at a time. In the machine method a stack of sieves is shaken in one operation. As it is not practical to measure the rate of passage of material through the individual sieves, the stack is shaken for a period of time long enough to insure that each particle has found its proper place.

It has been found that for some materials the agreement between hand and machine sieving is very good, whereas for other materials it is very poor. There is considerable doubt about the absolute accuracy of either method, but hand sieving is usually recommended because it is more reproducible. Machine sieving is much less time consuming and easier and, hence, if it were as reproducible as hand sieving, it would be preferred.

1. Rate of Sieving

The progress of sieving can be followed by weighing the material remaining on the sieves after convenient measured intervals of time, usually 1 to 2 min. The weight of material on the sieves is thus determined as a function of time. A graphical representation of this relationship is logically called a "sieving curve" and is illustrated in figure 6 for the different sieves in a stack.

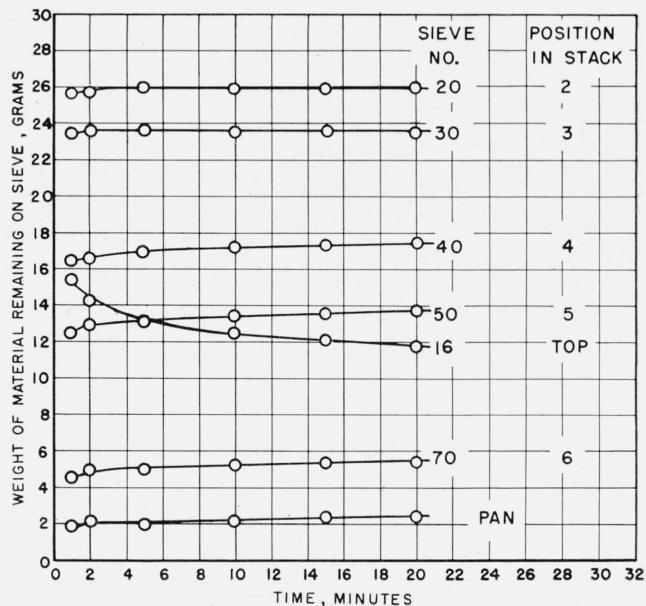


FIGURE 6. Typical sieving curves (weight retained as a function of time) for bone char 32.

¹⁰ The stopping point for sieving is explained in detail in section III, 2.

At the start all the material is placed on the top sieve, and this sieve can only lose weight as the sieving progresses. The weight of material on all the other sieves is determined by the rate at which material falls upon it from above and leaves it by passing through. Each of these rates is called a sieving rate. The weight on the intermediate sieves sometimes decreases and sometimes increases, depending upon which of these sieving rates is larger. If they are approximately the same for each sieve, then the weight remaining on intermediate sieves remains constant and only the top sieve and pan will show any change, as may be seen in figure 6. Thus, the rate of change observed on the top sieve and in the pan are true sieving rates, whereas the rates of change on intermediate sieves are differences between sieving rates.

In order to study sieving rates it is quite obvious that, at most, only two sieves can be shaken at a time. The material on the top sieve and the material in the pan are weighed every few minutes to determine the two sieving rates. For practical purposes it is far better to shake only one sieve at a time. The sieving rate can be obtained from the weight of material falling into the pan, and in this way the material on the sieve is not disturbed during the weighing.

A typical sieving-rate curve is shown in figure 7. It is seen that the rate drops off very rapidly at first, but then much more slowly, and was never observed to reach zero. This is further demonstrated in table 4, in which the shaking was carried on for 80 min,

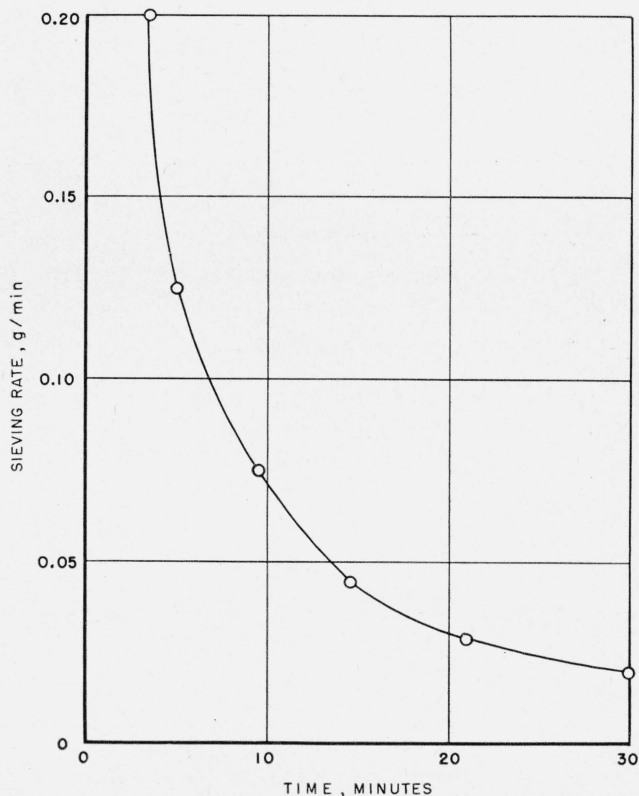


FIGURE 7. Typical sieving rate curve for bone char.

TABLE 4. Effect of prolonged shaking on the Ro-Tap

Time of shaking	Weight on sieve	Time of shaking	Weight on sieve
<i>min</i>	<i>g</i>	<i>min</i>	<i>g</i>
2	19.0	15	16.3
3	18.3	20	16.0
4	17.8	36	15.1
5	17.7	50	14.6
7	17.0	60	14.5
9	16.8	70	14.3
11	16.7	80	14.0
13	16.4		

and the weight of bone char on the sieve was still decreasing. The continued passage of material after such long periods is due to attrition of the particles. Because of this attrition, it is desirable to shake the sieves for a short time only.

2. End Point of Sieving

The end point of sieving would be attained when no more material passed any of the sieves in the stack, or when all sieving rates became zero. However, as has been shown, the sieving rate never reaches zero, and therefore a definite end point of sieving is not attainable. It is necessary, therefore, to employ another well-defined and reproducible point that may serve as a close approximation. A designated small value of the sieving rate serves this purpose. In order that the end point be reproducible the sieving rate must be very low, so that if shaking is carried on a minute or so too much or too little, the change in the quantity on the sieve is insignificant. The magnitude of an insignificant change depends upon the required accuracy. A few tenths of 1 percent of the weight of the sample is sufficient reproducibility for many purposes.

The standard sieving methods of the ASTM for cement [12], powdered coal [14], and paving materials [16] require that the terminal rate be 0.05 g/min, and for refractories [15], and soap [13] a terminal rate of 0.1 g/min. For nongranular roofing materials [10], the rate is 0.05 percent of the weight of the total sample per minute, whereas for fine and coarse aggregates used in concrete [17] it is 1 percent of the residue on the sieve per minute. A terminal rate expressed as a percentage of the sample insures that the same accuracy is obtained for a sample of any size. However, since it can be shown that sieving rate is nearly independent of weight of sample, the terminal rate is better expressed in terms of the sieving rates.

A suitable stopping point for bone char was found to be a sieving rate of 0.1 g/min through a standard 8-in.-diameter sieve. A lower value was not suitable because of the abrasion of some soft chars. For this rate to be of the order of magnitude of 0.1 percent/min, a sample of about 100 g should be used. The same stopping point was used for both hand and machine sieving. When shaking by hand, the sieves were tapped and shaken about 100 to 130 times per min, and as the end point was approached, the amount passing was weighed each minute.

TABLE 5. *Reproducibility of hand sieving during repeated analyses of the same sample of char*

Sieve fraction	Repeated sieve analyses			Mean	Standard deviation from the mean
	1	2	3		
CHAR 69 B GRAB					
	%	%	%	%	%
On 12	2.3	2.4	2.3	2.3	0.071
12 to 16	12.8	12.9	12.8	12.8	.071
16 to 20	22.7	22.6	22.9	22.7	.158
20 to 30	25.1	25.3	25.1	25.2	.123
30 to 40	18.7	18.6	18.6	18.6	.071
40 to 50	12.4	12.4	12.3	12.4	.071
Through 50	6.0	5.8	6.0	5.9	.123
				Avg...	0.098
CHAR 32 GRAB					
On 16	4.3	4.4	4.4	4.4	0.071
16 to 20	20.9	20.9	20.8	20.9	.071
20 to 30	25.3	25.2	25.0	25.2	.158
30 to 40	20.0	19.6	20.0	19.9	.235
40 to 50	17.3	17.5	17.5	17.4	.123
50 to 70	8.0	8.1	8.0	8.0	.071
Through 70	4.2	4.3	4.3	4.3	.071
				Avg...	0.114

Several repeated analyses with hand sieving are given in table 5. It is seen that the standard deviation from the mean is approximately 0.1 percent of the weight of the total sample.

The time required to reach the terminal sieving rate of 0.1 g/min on a Ro-Tap machine is called the sieving time. It is determined by the point at which the slope of the sieving curve becomes 0.1 g/min.

Fagerholt [2] derived a purely theoretical mathematical expression for the relation between the weight (W) of material remaining on a sieve and the time of shaking (t).

$$W = W_{\infty} + \frac{C}{\sqrt{t}}$$

where W_{∞} is the weight that would remain after an infinite time, and C is a constant.

In his derivation it was necessary for Fagerholt to assume that (1) the rate of passage is proportional to the weight that can pass the sieve, (2) probability of passing depends upon size of particle relative to size of opening, (3) particle size distribution is constant in the range near the sieve opening, (4) sufficient time has elapsed for the passage of particles very much smaller than the sieve opening, and (5) the sieve openings are absolutely uniform in size.

It is shown in section IV, 5, that Fagerholt's second assumption is not strictly correct, because the probability of passing is also dependent upon the relative motion of particle and opening. The fifth assumption is seldom realized even for new sieves. The nonuniformity may be very great for old, distorted sieves (see fig. 3).

The rate of passage of material through the sieve is obtained by taking the derivative of the above expression.

$$\frac{dW}{dt} = -\frac{C}{2} t^{-1.5}$$

When the rate of passage of material through a sieve is plotted against time of shaking on log paper, a straight or very slightly curved line results (fig. 12) with a slope between -1.1 and -1.7 . The failure to achieve always a slope of -1.5 can be attributed to nonconformity to the assumptions used in the derivation, especially the second and fifth. Nevertheless, the plot does serve as a convenient means to determine the sieving time.

3. Ro-Tap Machines

There are two types of Ro-Tap machines in use today. The older model has two eccentrics, whereas the model being produced today has an eccentric on one side and a reciprocating motion on the other. The older model thus gives a rotary motion in the horizontal plane, and the newer model gives a combined rotary and reciprocating motion. In both machines the ratio of rotations to taps is equal to 1.875. The substance upon which the knocker strikes can be varied, and commonly used materials are cork, rubber, and hardwood.

To obtain some idea of the performance of various Ro-Tap machines, samples of two different service chars were sieved in seven different Ro-Taps and by hand. The same samples were sieved repeatedly to eliminate any sampling error. The magnitude of the attrition was found to be negligible by making the first and last analysis on the same Ro-Tap machine under identical conditions (see table 6). The same set of sieves was used by the same operator in all cases. The shaking was continued for 10 min for all tests except hand sieving. The Ro-Tap machines tested were in daily use, testing various materials and were used as found without alteration in any way. The characteristics of the various machines and the sieve analyses are recorded in table 6. The results of machine No. 1 are in closest agreement with hand shaking in these cases. However, this is not always the case, as will be shown later in the section on speed of Ro-Tap.

The coefficient of variation for the top sieve was found to be 23 percent, whereas for all others it ranged from 2 to 6 percent. This is because the weight on the top sieve can only decrease, whereas on all other sieves material falls through about as fast as it falls upon the sieves. The weight in the pan can only increase. Thus, if one shaking method causes more particles to pass the sieves than another, the top sieve and pan should indicate the greatest difference with very little difference on the intermediate sieves. An examination of table 6 shows that this is what happens, except that a large variation did not appear in the pan. Apparently, the variation that should have appeared in the pan was absorbed by the intermediate sieves.

4. Variation Due to Ro-Tap Knockers and Position of Sieves in Stack

It was decided to test the two types of Ro-Tap machines having one or two eccentric bearings with various knocker cushions and various chars on each.

TABLE 6. Variation among Ro-Tap machines

Machine	1	2	3	4	5	6	7	* 1	Hand	Mean of 7 machines, <i>m</i>	Standard deviation from mean, σ	Coefficient of variation, σ/m
Speed (taps/min)	110	155	155	160	160	158	156	112				
Eccentrics	2	2	1	1	2	1	1	2				
Knocker	Rubber	Rubber	Cork	Rubber	Rubber	Maple	Cork	Rubber				

CHAR 32

On sieve No.:	%	%	%	%	%	%	%	%	%	%	%	%
16	4.9	5.6	5.6	5.9	8.6	4.6	5.2	4.7	4.3	5.77	1.325	23.0
20	21.6	22.9	22.5	22.4	22.3	22.3	22.4	21.4	20.9	22.34	.387	1.7
30	25.0	25.1	24.2	24.2	24.0	24.8	24.8	25.0	25.3	24.59	.441	1.8
40	19.7	20.0	19.4	19.3	19.2	19.5	19.4	19.8	20.0	19.50	.271	1.4
50	16.8	15.3	16.5	16.2	15.1	16.8	16.5	17.0	17.3	16.17	.697	4.3
70	7.8	7.4	7.7	7.8	7.1	7.7	7.6	7.8	8.0	7.59	.255	3.4
Pan	4.2	3.7	4.1	4.2	3.7	4.3	4.1	4.3	4.2	4.04	.244	6.0

CHAR 69 B

12	2.5	3.9	2.8	3.4	4.5	2.7	2.7	2.3	2.3	3.21	0.750	23.4
16	13.5	14.7	14.0	14.2	15.4	13.6	14.0	13.1	12.7	14.20	.661	4.6
20	22.7	22.0	22.7	22.5	22.4	23.0	22.6	22.9	22.9	22.56	.310	1.4
30	25.3	25.2	24.6	24.5	24.4	24.7	24.9	25.4	25.1	24.80	.347	1.4
40	18.4	17.9	18.2	18.0	17.5	18.3	18.2	18.4	18.6	18.07	.304	1.7
50	12.0	11.0	12.1	11.8	10.7	12.0	12.0	12.2	12.3	11.66	.565	4.8
Pan	5.6	5.3	5.6	5.6	5.1	5.7	5.6	5.8	6.0	5.50	.216	3.9

* Included as a check for attrition.

TABLE 7. Weight of material remaining on sieves for the 16 experimental variations

	Knocker <i>K</i> ₁	Knocker <i>K</i> ₂	Knocker <i>K</i> ₃	Knocker <i>K</i> ₄
TOP SIEVE				
	<i>g</i>	<i>g</i>	<i>g</i>	<i>g</i>
Machine 1 with char 1	21.1	21.0	20.8	22.8
Machine 2 with char 2	15.8	15.9	15.6	16.0
Machine 2 with char 1	21.4	21.4	21.1	21.5
Machine 1 with char 2	15.0	14.9	15.2	16.9
SECOND SIEVE				
Machine 1 with char 1	17.0	17.3	17.1	17.7
Machine 2 with char 2	18.7	18.6	18.6	18.6
Machine 2 with char 1	17.2	17.3	17.4	17.3
Machine 1 with char 2	18.6	18.7	18.6	18.4
THIRD SIEVE				
Machine 1 with char 1	16.6	16.1	16.6	15.4
Machine 2 with char 2	14.7	14.7	14.8	14.6
Machine 2 with char 1	16.1	16.0	16.1	16.0
Machine 1 with char 2	14.9	14.9	14.7	15.3
PAN				
Machine 1 with char 1	16.2	16.2	16.2	14.8
Machine 2 with char 2	15.7	15.6	15.8	15.5
Machine 2 with char 1	16.0	15.9	16.1	15.9
Machine 1 with char 2	16.3	16.3	16.3	14.2

The experiments were arranged to give the greatest amount of information by application of statistical methods of evaluation.¹¹ Two different service chars were used on two different machines. The use of three different knocker cushions and the omission of the knocker made four different variations of knockers. Three sieves and a pan were used. The time for each shaking was 7.5 min. A tabulation of the variations employed is given below:

¹¹ The authors are indebted to John Mandel for aid in the design of the experiments and their evaluation by statistical methods.

Machines:

- M*₁—2 eccentrics at 115 taps/min.
- M*₂—1 eccentric at 150 taps/min.

Chars:

- C*₁—70.7 g of char 32 with sieves No. 18, 25, 35, pan.
- C*₂—64.8 g of char 33 with sieves No. 20, 30, 40, pan.

Knocker cushions:

- K*₁—Rubber.
- K*₂—Cork.
- K*₃—Hardwood (maple).
- K*₄—None.

Experiments were undertaken with combinations of each of the possible machines, chars, or knocker cushions by using the sieves designated for each char. To eliminate any possible bias due to attrition of the char, the order of the experiments was taken from a Latin square arrangement. The Latin square is a statistical method of removing bias due to the order of experiment, as shown here.

Order of experiments

	Knocker <i>K</i> ₁	Knocker <i>K</i> ₂	Knocker <i>K</i> ₃	Knocker <i>K</i> ₄
Machine 1 with char 1	1	2	3	4
Machine 2 with char 2	6	5	8	7
Machine 2 with char 1	11	12	9	10
Machine 1 with char 2	16	15	14	13

The results for each sieve are given in table 7. From an analysis of variance (see appendix), it was found that the variation due to knockers, chars, and the interaction between machines and knockers

is highly significant and the interaction between machines and chars is significant. Table 7 shows that the absence of a knocker (condition knocker 4) is the greatest cause of the variation. This means that sieve analyses obtained without the knocker are not comparable to those in which a knocker is used. When the analysis of variance is repeated omitting K_4 (see appendix), it is found that there is significant variation between chars on all sieves and that there is significant variation between machines in the case of the top sieve and pan. There is no significant variation in any other case.

That a difference was found between chars was to be expected. The important point is that there is a difference between machines but none among knockers nor any interactions among combinations of knockers, chars, or machines. It was found that the magnitude of the difference between machines was dependent upon the speed of the mechanism, and this point is covered further under that heading. That no significant difference was found among knockers indicates that this is not an important source of error in machine sieving and need not be rigidly controlled. The fact that the interactions with chars were not significant indicates that all chars react in the same way and that a sieving procedure that will produce good results for one char will also produce good results for other chars.

Some of the older machines have flat leaf springs to hold the sieves at the bottom instead of the cast iron plate, as in the newer models. It was found that the tension in these springs had no effect upon the sieving.

When materials of different grist are sieved, it is expedient to have a certain sieve sometimes at the top of the stack and sometimes at the bottom. To determine whether or not a sieve gives the same results in the various positions, samples of service char were sieved on a single sieve at various positions in the stack. All the other sieves that filled up the stack were very much larger, and no material was retained on any of them. It was concluded that the position of the sieve in the stack is of no importance, as far as the results on that one sieve are concerned.

5. Speed of Ro-Tap

Early experiments showed that the speed of the Ro-Tap has an important effect on the final weight of material that remains on a sieve, and that this effect depends upon the size of opening. Therefore, a series of experiments was undertaken in which sieving curves were determined for closely sized fractions of char at various speeds of the Ro-Tap. A weight of 15.00 g of each of the following char fractions was placed on the sieve designated below, and the sieving repeated at various speeds. A fraction of char previously sieved to pass No. 12 and be retained on No. 16 was sieved on No. 14; similarly, a 30 to 40 fraction was sieved on a No. 35; and a 70 to 100 fraction on sieve No. 80. The general shape of the sieving curves is the same for all sizes, and typical results are shown in figure 8. It is noted that the curve at the high speed (150 taps/min) levels

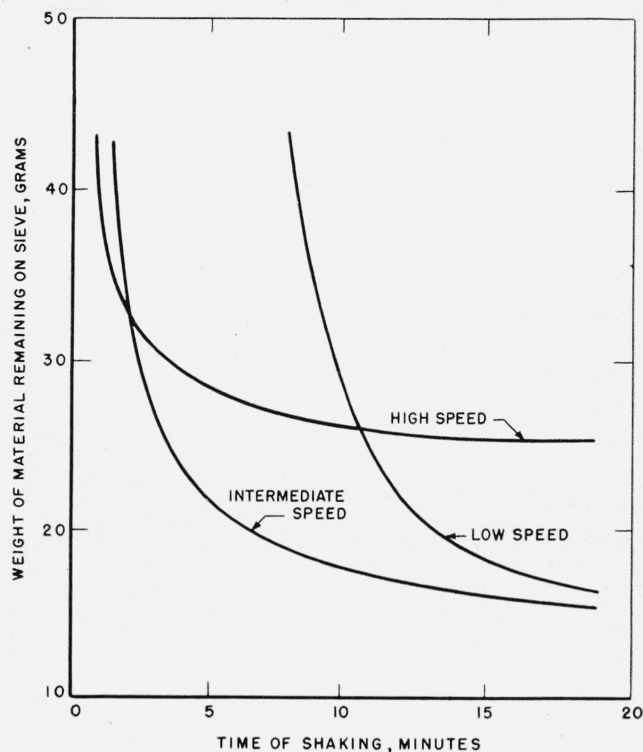


FIGURE 8. Effect of Ro-Tap speed on weight retained as a function of time.

off much higher than the curve for intermediate speeds (115 taps/min), whereas the curve at low speeds (less than 90 taps/min) levels off at the same value as that for intermediate speeds but takes more time to do it.

This effect is brought out more clearly in figures 9 and 10. The weight of material on a sieve after 10 min of shaking is plotted as a function of speed in figure 9. In figure 10 the weight of material remaining on the sieve when the sieving rate falls to 0.1 g/min (termination of sieving) is plotted as a function of speed and compared to hand shaking. In figure 9 it is seen that there is a definite minimum in the amount remaining on the sieves at a speed of about 115 taps/min; the curves in figure 10 merely flatten out at low speeds. This means that at high speeds (150 taps/min) the sieve retains some particles that pass through it at lower speeds. At 115 taps/min the minimum amount is held on the sieve, and further reduction of speed only serves to lengthen the time required to complete the sieving.

Since it is desirable to have the sieve analysis give the closest possible representation of particle size distribution, it follows that the most accurate sieve analysis will be obtained at the speed at which the minimum occurs, namely 115 taps/min. Also, sieve analyses made at the speed of the minimum should be more reproducible, because adjacent to the minimum the curves slope very slightly. Thus, if the speed of Ro-Tap varies by a few taps a minute, then the effect on the amount passing the sieve is minimized. At the usual speed of 150 taps/min, the

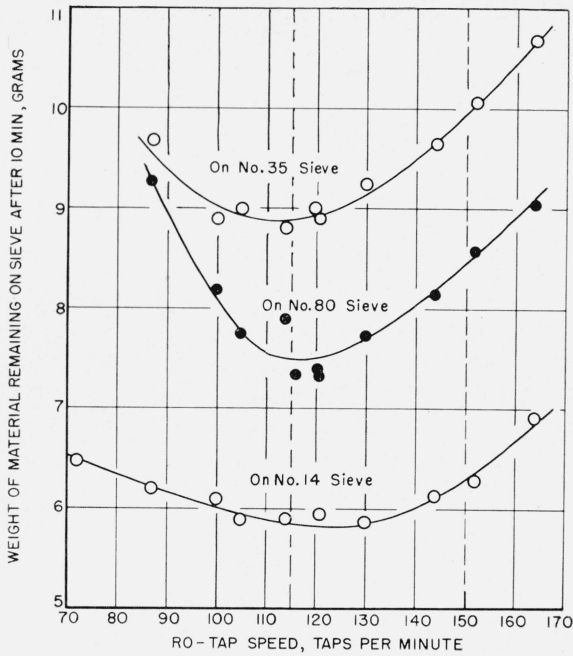


FIGURE 9. Effect of Ro-Tap speed on the weight of bone char retained on various sieves after 10 minutes.

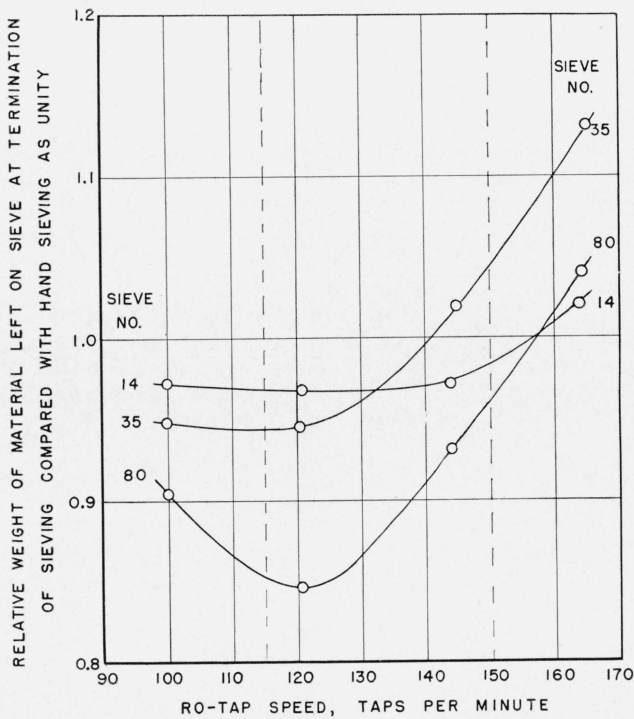


FIGURE 10. Effect of Ro-Tap speed on the weight retained on various sieves at the termination of sieving compared with hand sieving as unity.

slopes of the curves in figures 9 and 10 are such that a variation of 5 taps/min changes the amount remaining on the sieve by about 2 percent. The variations noted in table 6 are largely due to variations in speed. The reproducibility of sieve analyses

made at one constant speed is essentially the same for all types of machines regardless of the speed.

The agreement between hand and machine sieving at a speed of 150 taps/min is somewhat better than at a speed of 115 taps/min. However, the improved reproducibility and the apparent greater accuracy of machine analyses at 115 taps/min more than outweighs the disadvantage of nonagreement with hand sieving for materials such as bone char.

To study the agreement of the two types of Ro-Tap machines at various speeds, three samples of char having different particle sizes were run on both types. The weights on the sieves after 10 min of sieving are shown as a function of speed in figure 11. The results are in substantial agreement for all particle sizes at speeds below 115 taps/min. At higher speeds the results obtained on the two machines become more divergent. The two-eccentric machine, in which the motion is more violent, does not pass as much material as the one-eccentric machine per unit of time.

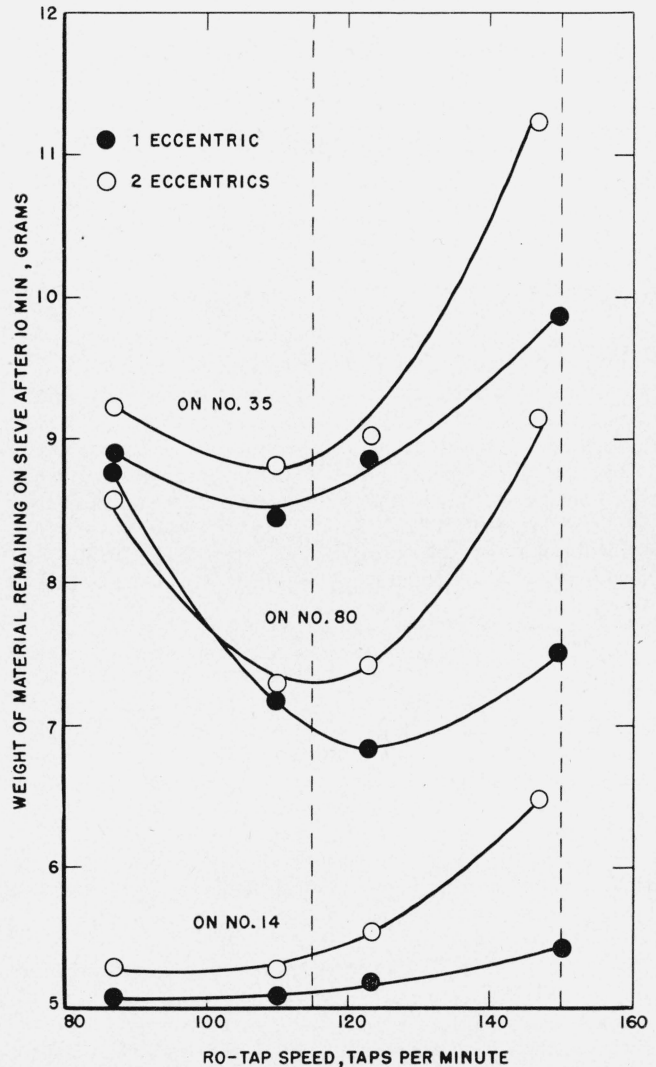


FIGURE 11. Effect of Ro-Tap speed with two types of machines.

Fahrenwald and Stockdale [19] studied the effect of the motion of the sieve. They found that the highest rates of transport of material through the sieve could be obtained at very high speeds of shaking if the amplitude of motion was about the same as the size of the sieve openings. The amplitude of motion of the Ro-Tap machine is about 1 in., which is much larger than the opening of any sieve studied here. Apparently the selective retention of *slightly undersized particles* at high speeds of the Ro-Tap is to be attributed to excessive amplitude of motion in the horizontal plane. The enhanced retention of *all particle sizes* at low speeds is due to the lack of sufficient motion. It is possible to express the probability, P , of a particle passing through a sieve opening in terms of fundamental mechanics. Each particle can be uniquely located in the space above the sieve by six coordinates, three of translation and three of momentum. The probability, P , is then a ratio, in which the denominator is the product of all possible positional and momentum values in phase space, and the numerator is the particular positional and momentum values required for the particle to pass the opening. All types of sieving devices that utilize forced vibrations should be subject to this analysis and exhibit an optimum value of frequency for maximum transport through the sieve openings.

6. Effect of Speed of Ro-Tap for Various Materials

In order to determine whether the effect of variations in speed was peculiar to bone char or general for all types of material, a number of different materials, particle shapes, and particle sizes were examined. The results are summarized in table 8. The weight of material remaining on the sieves varied over wide ranges, but since the actual weight is not important the amounts remaining on the sieves are expressed relative to the minimum amount. The data should be regarded as qualitative. The fact that one material shows a 10-percent and another a 50-percent increase in the amount retained on the sieve at high speed is of no significance; it only reflects the different particle size distributions. The

same behavior with regard to speed was found for all the materials tested; that is, at a speed of Ro-Tap of approximately 150 taps/min some under-sized particles are retained that will pass through at a speed of approximately 115 taps/min.

IV. Sample for Sieve Analysis

It is of importance that the sample for sieve analysis be representative of the material from which it was taken. However, this study is primarily concerned with the influence of weight and other physical properties of the sample on the sieving. The sample for sieve analysis is usually only a small fraction of the original material and must be prepared by coning and quartering or the use of a riffle, or the use of other suitable sample divider. The entire end product of the sample-reduction process must be used as the sample for the sieve analysis, otherwise it would completely defeat the purpose of the reduction process.

1. Weight of Sample

The minimum number of particles that can be considered a representative sample of a heterogeneous material such as bone char is about 10,000, and it would be preferable to have many more. From this point of view, the sample for sieve analysis should be large enough to have this number in each fraction. Ten thousand particles of 12 to 14 fraction of bone char weigh about 30 g, and 10,000 particles of 80 to 100 fraction weigh about 30 mg.

The testing sieves are only 8 in. in diameter, and if they are loaded too heavily a condition known as "blinding" occurs. When this takes place, nearly all the openings become plugged by particles wedged into them, and even material very much finer than the sieve opening cannot pass. Too great a depth of material on the sieve and the hammering of the oversize pieces into the openings by the many pieces above contribute to the blinding. It is generally agreed that the ideal depth of material on the sieve is no more than one or two particles. A layer two particles deep of 80 to 100 fraction of bone char with a bulk density of 60 lb/ft³ on an 8-in. sieve weighs about 10 g. A layer of 10 to 12 fraction of material this deep weighs 100 g. A layer two particles deep contains more than 10,000 particles for sieves finer than No. 8. It has been found that material four or six particles deep can be successfully sieved, but more than this causes excessive blinding.

In the range of particle sizes commonly found in granular materials, a convenient weight of sample is 100 g. Samples of bone char larger than 500 g always produce excessive blinding of the sieve, and samples smaller than 50 g always give very erratic results, because the coarser fractions contain too few particles. Since it is not practicable to prepare a sample of exactly 100.00 g by a sample reduction process, approximately 100 g must be used. The effect of sample size must be determined so that sieve analyses made with different sized samples can be reduced to a comparable basis. Preliminary ex-

TABLE 8. Effect of Ro-Tap speed for various materials

Material	Particle shape	U. S. Standard Sieve No.	Relative amount on sieve	Speed
Glass	Beads	25	1.08	88
			1.00	116
			1.08	150
Do	do	6	1.37	91
			1.00	120
			1.38	152
Char 32	Granular	50	1.00	118
			1.09	155
			1.00	122
Char 68 C	do	8	1.52	158
			1.00	122
			1.04	156
Bismuth	do	50	1.00	122
			1.00	122
			1.08	156
Iron	Filings	30	1.00	122
			1.08	156
			1.00	117
Sand	Granular	25	1.25	156
			1.00	117
			1.00	117
Floridin (73 H)	Rounded	40	1.00	117
			1.13	156

periments showed that identical sieve analyses could be obtained with samples from 50 to 500 g if the shaking was continued just long enough to reach the end point of sieving.

It might be expected that the sieving time required to reach this end point would be directly proportional to the weight of sample. This is found to be true for any one particular sample. However, further investigation showed that the sieving time was also dependent upon the particle size distribution, particle size, the particle shape, and sieve uniformity.

2. Effect of Particle Size Distribution

An experiment was performed to study the influence of those particles that are of such sizes that they might hinder the passage of the particles that are a near fit. A service bone char (char 32) was carefully sieved into the following fractions:

Fraction	Weight
Through No. 25 on No. 30	90
Through No. 30 on No. 35	10
Through No. 35 on No. 40	10
Through No. 40 on No. 45	90

First, the 30 to 35 and 35 to 40 fractions (total weight 20 g) were mixed together and sieved on the No. 35 sieve and the rate determined as a function of time. Then the 25 to 30 fraction was added (total weight 110 g) and the sieving repeated; finally, all four fractions were mixed (total weight 200 g) and again the sieving repeated. The results are given in table 9 and shown graphically in figure 12. It is at once apparent that the same value for sieving

time was obtained for all three experiments, although the weight of the sample varies tenfold, and that many over-sized particles do not effect the passage of the near-fit particles. The near-fit particles are defined as those that pass the next larger sieve and are retained on the next smaller sieve in $\sqrt{2}$ series. The effect of sieve uniformity does not enter here, because only one No. 35 sieve was used. From these experiments it can be concluded that the sieving time is not affected by the weight of the entire sample nor by the weight of that portion that remains on top of the sieve, but rather by the weight of the near-fit particles.

TABLE 9. Effect of size of sample and relative particle size distribution on the sieving time of fractions of char 32

Time interval	Additional weight through	Weight rate	Mean time
10 g (30 to 35)+10 g (35 to 40) on No. 35			
<i>min</i>	<i>g</i>	<i>g/min</i>	<i>min</i>
2 to 3	0.25	0.25	2.5
3 to 4	.20	.20	3.5
4 to 6	.25	.125	5
6 to 10	.24	.060	8
10 to 17	.30	.043	13.5
17 to 25	.23	.029	21
25 to 35	.15	.015	30
90 g (25 to 30)+10 g (30 to 35)+10 g (35 to 40) on No. 35			
2.5 to 3.5	0.16	0.16	3
3.5 to 6	.33	.132	4.75
6 to 10	.34	.085	8
10 to 16	.36	.060	13
90 g (25 to 30)+10 g (30 to 35)+10 g (35 to 40)+90 g (40 to 45) on No. 35			
2.16 to 3	0.25	0.30	2.58
3 to 5	.37	.185	4
5 to 9	.32	.08	7
9 to 15	.37	.062	12

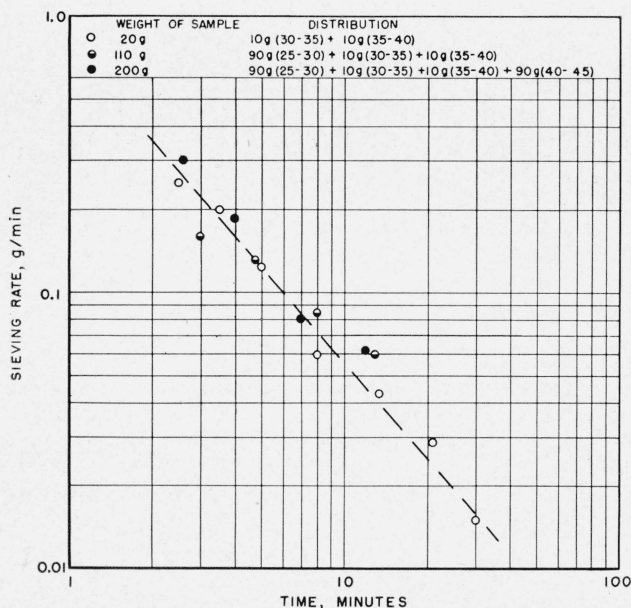


FIGURE 12. Effect of size of sample and particle size distribution on sieving time.

3. Effect of Particle Size on Sieving Time

The near-fit material has to fall upon the openings a large number of times before it is known whether or not it passes. Accordingly, the sieving time should be dependent upon the number of near-fit particles per sieve opening. The number of particles (N) is proportional to the weight of material (W) and inversely proportional to the cube of the diameter of the particle (d), ($N=k_1 W/d^3$). The number of sieve openings (H) in a constant sieve area is inversely proportional to the square of the diameter of the opening,¹² ($H=k_2/d^2$). In these relationships k_1 and k_2 are constants. Thus, the number of particles per sieve opening is given by the following relationship: $N/H=(k_1/k_2) \times (W/d)$. Hence, the sieving time should be dependent upon the weight of near-fit material divided by the size of the sieve opening.

To check this hypothesis, experiments were conducted with sieves of certified uniformity in which

¹² More exactly, the number of openings is proportional to the free or open area, but the wire diameters and sieve openings are such that the ratio of open area to total sieve area is about the same for all sieve sizes.

the weight of near-fit particles was varied over wide limits for several different sieve sizes and particle shapes. The sieving times are given in table 10. In figure 13 the sieving time is plotted as a function of the weight of near-fit particles divided by the size of the opening. Sizes equal to or smaller than

TABLE 10. Sieving times for various weights, sizes, and shapes of particles

Material	Weight of near-fit, W_N	Sieve No.	Diameter of opening, d	Sieving time	W_N/d
	<i>g</i>		<i>cm</i>	<i>min</i>	<i>g/cm</i>
Chars.....	40	8	0.238	11	168
Do.....	500	16	.119	115	4,200
Do.....	200	16	.119	48	1,680
Do.....	160	16	.119	37	1,345
Do.....	125	16	.119	32	1,050
Do.....	100	16	.119	25	840
Do.....	60	16	.119	16	504
Do.....	20	16	.119	6	168
Do.....	15	16	.119	4.5	126
Do.....	110	35	.050	33	2,200
Do.....	80	35	.050	21	1,600
Do.....	40	35	.050	9	800
Do.....	20	35	.050	8	400
Do.....	60	50	.0297	27	2,020
Do.....	40	50	.0297	21	1,346
Do.....	30	50	.0297	17	1,010
Do.....	10	50	.0297	5	337
Do.....	40	80	.0177	29	2,260
Do.....	25	80	.0177	19	1,412
Do.....	20	80	.0177	16	1,130
Do.....	10	80	.0177	8	565
Do.....	10	80	.0177	8.5	565
Do.....	11	270	.0053	32	2,070
Sand (angular).....	160	25	.071	45	2,250
Glass (spherical).....	100	25	.071	15	1,410
Do.....	25	25	.071	4	350
Floridin (round).....	35	50	.0297	11	1,170

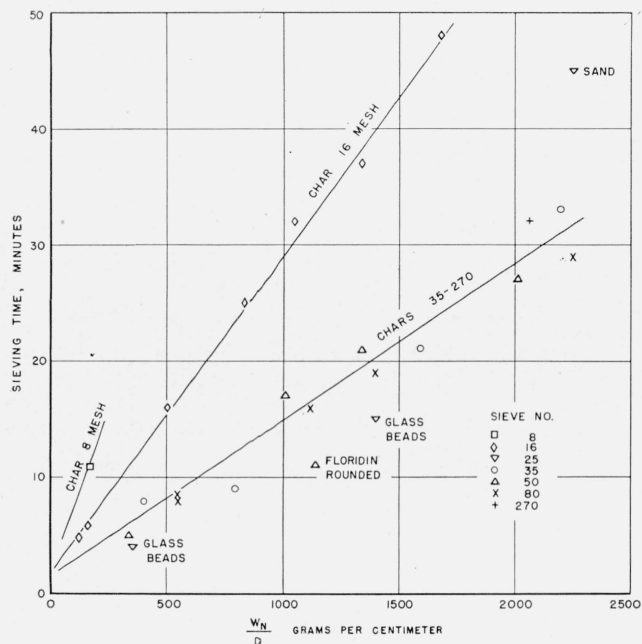


FIGURE 13. Correlation of sieving time with the number of near-fit particles per sieve opening.

The number of near-fit particles per sieve openings is proportional to W_N/d .

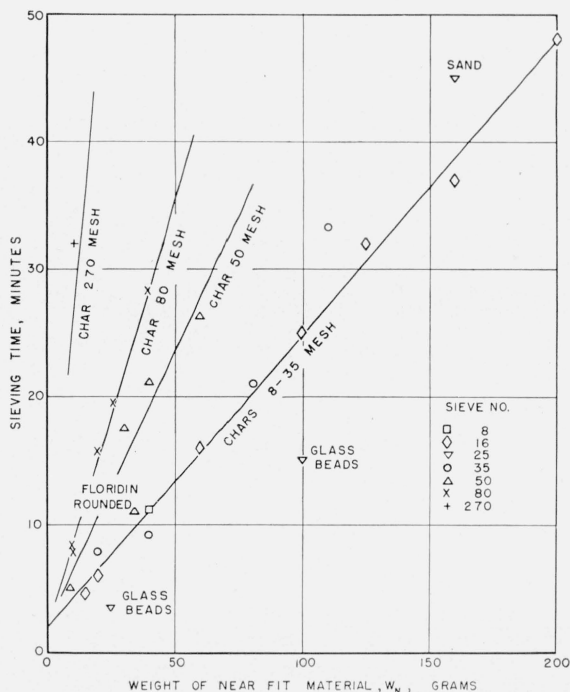


FIGURE 14. Dependence of sieving time on weight of near-fit material.

the opening of No. 35 fall near the same straight line, but sizes coarser than No. 35 fall near lines of increasing slope. If these sieving times are plotted as a function of the weight of near-fit particles (fig. 14), then sizes equal to or coarser than No. 35 fall near one line and those finer on lines of increasing slope. The reason for this behavior is not apparent. It may be noted from figures 13 and 14 that the round shapes require only about one-half as much sieving time as the irregular shapes. As all curves have an intercept of 1 or 2 min for zero weight of material, this is the time required for particles that are very much finer than the sieve to pass through^t

For bone chars and other granular materials of irregular shapes, the sieving time (T , min) can be represented as a function of the weight of near-fit material (W_N , g) and the sieve opening (d , cm) by the following expressions:

For sieves No. 35 and coarser:

$$T \text{ (min)} = 2 + 0.23 W_N$$

For sieves No. 35 and finer:

$$T \text{ (min)} = 1 + 0.0137 W_N/d.$$

The times should be calculated to the nearest minute. For rounded particles, somewhat less time is required with a minimum of one-half of the values obtained from these equations. The time of shaking of a stack of sieves should be determined by the sieve requiring the maximum time. In the usual sieving operation the approximate sieve analysis is not previously known, so that there is no way of determining the sieving time until a trial run is completed. For the trial run, 10 min is a suitable shaking time for samples of about 100 g.

It has been noted that when sieving service bone chars with the $\sqrt{2}$ series of sieves, there is usually a maximum of 20 to 35 percent of the total weight (W) of the sample on any one sieve. Hence, the weight of near-fit W_N on that sieve is to a first approximation about three-tenths of the total sample, namely, $W_N = 0.3 W$. The approximate sieving time for service chars can then be simply ascertained for a maximum occurring on a sieve coarser than No. 35:

$$T(\text{min}) = 2 + 0.07 W,$$

and for a maximum occurring on a sieve finer than No. 35:

$$T(\text{min}) = 1 + 0.004 \frac{W}{d}$$

It is recommended that a sample weighing not more than 150 g nor less than 100 g be used and the sieving time be taken from the above relations, which are expressed graphically in figure 15. If a maximum of

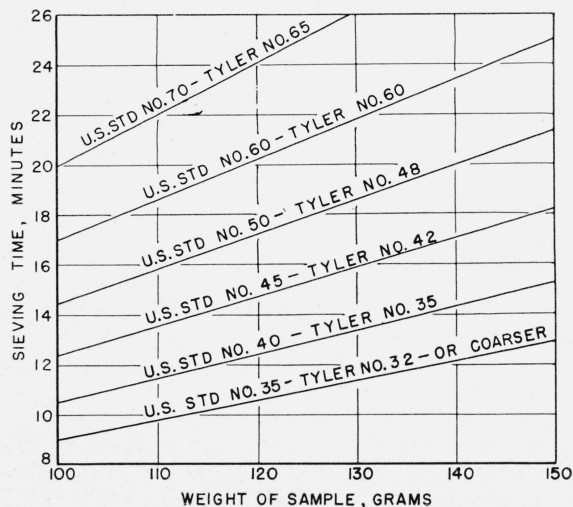


FIGURE 15. Approximate sieving time as a function of the weight of the bone char.

This method is to be used only if a maximum of 20 to 35 percent of the sample is retained on the sieve.

more than 35 percent or less than 20 percent be retained on any sieve, or if the sieves used are not the $\sqrt{2}$ series, or, if it is necessary to use less than 100 g or more than 150 g, then figure 15 and the approximate equations do not apply. It is then necessary to use the more exact relations given in the text. For example, assume that a preliminary sieve analysis showed that a certain char dust was about 42 percent between the No. 50 and 70 sieve and about 34 percent between the No. 70 and 100 sieve. A new sample, which weighed 108 g, was prepared on the riffle. As the sieves are in the $\sqrt{2}$ series, the weight on the sieves is approximately equal to the weight of near-fit material. Hence the weight of near-fit expected on the No. 70 sieve is $0.42 \times 108 = 45.4$ g and that on the No. 100 is $0.34 \times 108 = 36.8$ g. The open-

ing of the No. 70 sieve is 0.021 cm and that of the No. 100 is 0.0149 cm. From the relationship given for sieves finer than No. 35, the sieving time for the No. 70 is

$$T = 1 + 0.0137 W_N/d = 1 + 0.0137 \times (45.4/0.021) = 31 \text{ min.}$$

For the No. 100 sieve

$$T = 1 + 0.0137 (36.8/0.0149) = 35 \text{ min.}$$

The sieving time for this sample is 35 min. The No. 100 sieve required the longest time, even though the maximum weight does not occur on this sieve.

4. Test for Sieve Uniformity

Having considered the influence on the sieving rate of particle size, particle-size distribution, weight of sample, method of shaking, and shaking time, it is possible to devise a test for sieve uniformity. Any correlation between sieving rate and sieve uniformity is necessarily approximate because of the many factors involved. It has been shown that the method of shaking has practically no effect on sieving rate as long as the speed of the Ro-Tap remains constant.

The material usually employed on the sieves can be used for testing for uniformity. A sample that gives the same sieving rate for all sizes of sieves may be prepared by adjusting the weight of near-fit material for each size sieve. Five minutes was found to be a suitable sieving time. The calculated weights of near-fit material required to give a sieving time of 5 min is given in table 11 for a service bone char. The weights of individual fractions needed for a test sample of a service bone char are listed in table 12. The sum of the weights of the sieve fractions immediately above and below any given sieve is equal to the required weight of near-fit material. As it has been shown that oversized and undersized particles do not affect the sieving rate, a master test sample can be made up consisting of the tabulated amounts of all fractions in the desired range of sieve sizes. If the entire $\sqrt{2}$ series of sieves is not available, then combined fractions can be used with little difference in over-all results.

In making a test, the prepared sample is placed on one sieve and shaken for intervals of 1 or 2 mins. After each period of shaking, the additional amount in the pan is weighed and the rate of passage of material through the sieves is plotted as a function of time of

TABLE 11. Calculated values of weight of near-fit material for a 5-minute sieving time for a service bone char

U. S. Standard Sieve No.	Weight of near-fit	U. S. Standard Sieve No.	Weight of near-fit
	<i>g</i>		<i>g</i>
35 and coarser	13.04	120	3.65
40	12.26	140	3.07
45	10.22	170	2.57
50	8.67	200	2.16
60	7.30	230	1.81
70	6.13	270	1.55
80	5.17	325	1.28
100	4.35	400	1.08

TABLE 12. Calculated weight of sieve fractions of a service bone char needed to obtain a test sample for sieve uniformity

Sieve fraction	Weight	Sieve fraction	Weight
30 to 35 and all coarser close fractions ^a	6.52 each	100 to 120	1.94
35 to 40	6.65	120 to 140	1.71
40 to 45	5.48	140 to 170	1.36
45 to 50	4.74	170 to 200	1.21
50 to 60	3.93	200 to 230	0.95
60 to 70	3.37	230 to 270	.86
70 to 80	2.76	270 to 325	.69
80 to 100	2.41	325 to 400	.59
		Through 400	.49

^a Includes the fractions 4 to 5, 5 to 6, 6 to 7, 7 to 8, 8 to 10, 10 to 12, 12 to 14, 14 to 16, 16 to 18, 18 to 20, 20 to 25, 25 to 30, and 30 to 35.

shaking. If the sieves are uniform the sieving rate drops to 0.1 g/min in 5 min. or less. Typical sieving rate curves for sieves of varying uniformity are shown in figure 16. The heavy line in figure 16 has been drawn to indicate an arbitrary dividing line between satisfactory and unsatisfactory sieves. It is significant that the sieving rates for nonuniform sieves are very erratic as compared with the regular behavior of satisfactory sieves. Any closer correlation be-

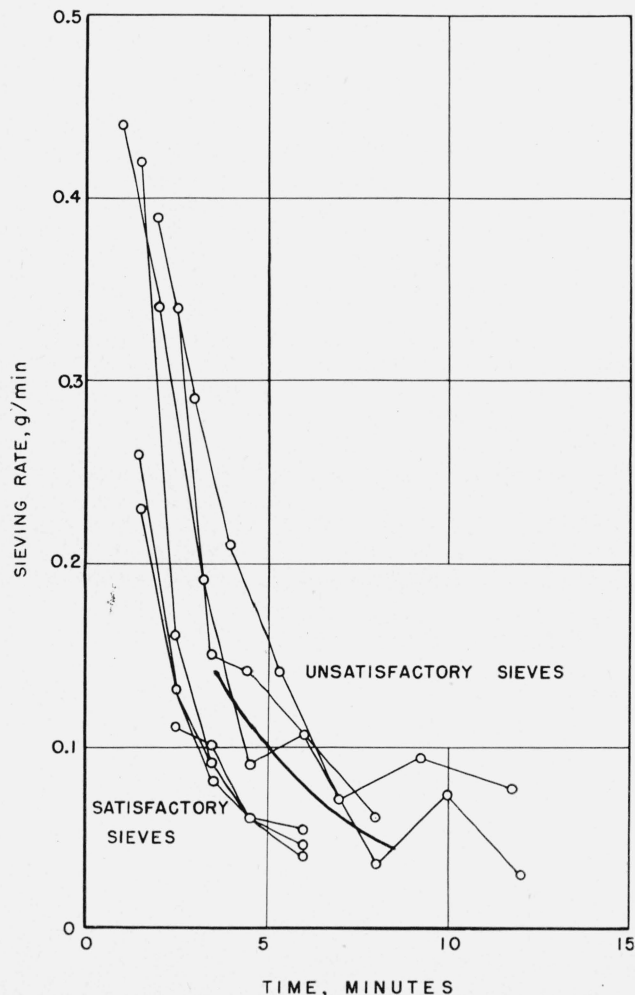


FIGURE 16. Suggested criteria for an approximate classification of sieves with reference to uniformity of openings.

tween sieve uniformity and sieving rate would require a long statistical study, which is not warranted at present.

5. Density and Hardness

Although substances ranging in density from about 1 g/cm³ (activated charcoal) to about 10 g/cm³ (bismuth) have been examined, no effect of particle density on sieving characteristics has been observed. The particle density for materials such as bone char and other adsorbents may be dependent upon particle size, thus complicating the relationship between the particle-size distributions by weight and by number.

As previously mentioned, material may continue indefinitely to pass through a sieve in motion because of the wearing of the particles. The minimum sieving rate, which is approached asymptotically, should be a measure of the abrasion resistance of the material. However, it would not be practical to determine abrasion resistance of bone char in this way, because it would require many hours of shaking on the Ro-Tap machine.

The resistance of the material to the type of abrasion encountered in the Ro-Tap machine can be measured very conveniently by repeating the sieve analyses several times. Any trend toward finer sizes is an indication of abrasion. In one experiment 128.6 g of a good service char was sieved five times for shaking periods of 10 min each. The weight of char remaining on the various sieves is plotted as a function of the number of sievings in figure 17. No particular trend is apparent on any sieve, but the weight in the pan definitely increases. Apparently, the wearing of bone char is such as to break off the sharp corners and thus produce fines rather than the fracture of large pieces. Attempts to detect changes on intermediate sieves fail because of the experimental error in sieving.

The rate of increase on the pan is 0.0034 g/min for the 128.6 g of sample, or 0.0026 percent/min. This rate of abrasion is quite insignificant in comparison with the other errors of sieving. The rate of abrasion of some soft chars is several times this value, and for a shaking period of 10 min, the increase in the pan fraction is sometimes as much as 0.1 percent. This is just large enough to be detected by a sieve analysis and, hence, very soft chars should be shaken as short a time as necessary.

6. Effect of Moisture on Sieve Analyses

The amount of moisture that a bone char can adsorb and still appear dry varies greatly with the char. A very good service char was found to adsorb about 17 percent of moisture (dry basis) before appearing wet, whereas a discard char adsorbed only about one-third that amount. The adsorption of water seems to be approximately proportional to the total surface area, the latter being measured by the adsorption of nitrogen at low temperature.

Small amounts of moisture have little effect upon passage of char particles through a sieve. It has been observed that bone char that appears to be dry

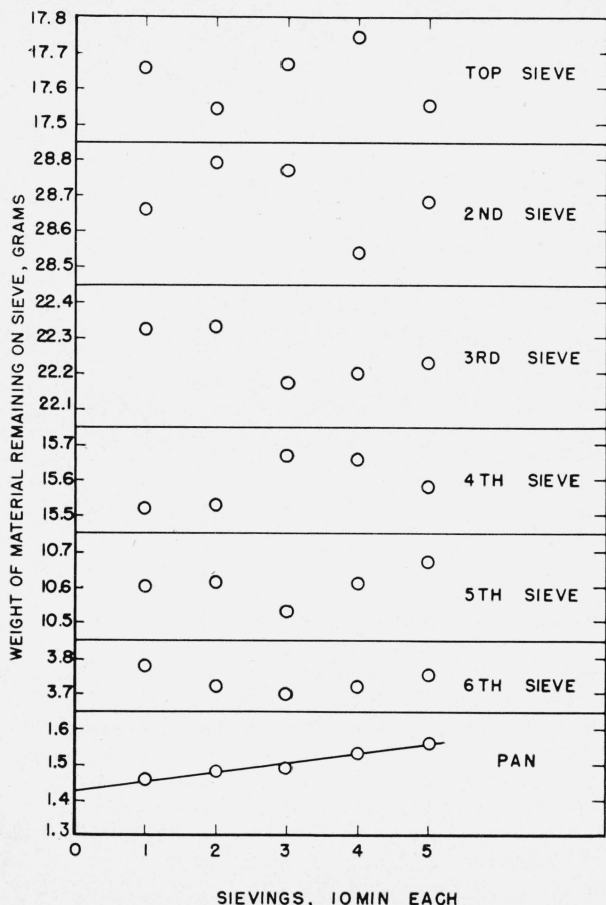


FIGURE 17. Attrition of bone char due to repeated sievings. The only significant trend is the increase in the weight of the pan fraction.

sieves just like dry material. On the other hand, if a bone char appears definitely to be moist, it does not sieve. In a test run, a sample of service char appearing wet, which contained a total moisture of 17 percent, was shaken for 10 min in a nest of six sieves in the usual fashion. It was observed that more than 70 percent of the sample remained on the top sieve and none had reached the bottom three sieves.

TABLE 13. Effect of moisture on sieve analysis of bone char

U. S. Standard Sieve No.	Sieve analysis dry char	Moisture content	Sieve analysis		Moisture content	Sieve analysis		U. S. Standard Sieve No.	Sieve analysis dry char	Moisture content	Sieve analysis		
			Wet	Dry (corrected)		Wet	Dry (corrected)				Wet	Dry (corrected)	
SERVICE CHAR 32 (GRAB SAMPLE)						SERVICE CHAR 34 (GRAB SAMPLE)							
			1.28% total moisture			13.6% total moisture						4.2% total moisture	
	%	%	%	%	%	%	%		%	%	%	%	
On 16	13.9	1.7	14.1	14.1	17.4	14.8	14.3	On 14	31.7	4.0	31.7	31.7	
16 to 20	36.4	1.3	36.2	36.2	13.8	36.7	36.6	14 to 16	26.9	4.8	27.9	27.8	
20 to 30	25.9	1.2	26.1	26.1	14.1	25.7	26.6	16 to 18	27.1	4.6	26.8	26.7	
30 to 40	15.0	1.1	14.9	14.0	11.7	14.6	14.8	18 to 20	9.65	3.4	9.18	9.26	
40 to 50	7.23	1.1	7.20	7.21	9.4	6.75	7.01	20 to 30	4.40	2.2	4.22	4.31	
50 to 70	1.38	0.7	1.36	1.37	5.2	1.25	1.35	Through 30	0.22	2.6	0.28	0.28	
Through 70	0.20		0.19	0.20		0.23	0.26						

The weights of moist char retained on the sieves will appear to be slightly different from that for the dried char, because the different particle sizes adsorb water to a different extent. Moist particles presumably would have the same size as when dry and, therefore, go through the same sieves; however, they would weigh more. Consequently, a sieve analysis of moist char must be accompanied by a moisture determination of the various fractions and the results calculated to a dry basis. The sieve analyses of a sample of chars 32 and 34 when dry and when containing different amounts of total moisture are given in table 13. A moisture determination was made for each fraction of the two wet chars and the sieve fraction corrected to correspond to dry char. When the char contains only 1 or 2 percent of moisture, the corrections are very small. Although a sieve analysis can be made with a char containing appreciable moisture, it is more practical to dry the complete sample ahead of time.

V. Summary and Recommended Procedure

It is not possible to assign a numerical value to the error to be ascribed to every particular source. It can be said, however, that the largest source of error by far is in the tolerances permitted in the average opening of testing sieves. When the standard $\sqrt{2}$ series of sieves conforming to present specifications is used, the differences when using different sets of sieves are usually in error by not more than 5 percent of the total sample. This error, due to the sieves, can be reduced to a few tenths of one percent of the total sample by determining the effective openings of the sieves with a prepared sample of spherical glass beads. However, nonuniform openings in the sieves produce erratic and inconsistent sieve analyses.

The method of shaking is a secondary source of error, in general, as long as some uniformity is observed. The position of the sieve in the stack, the knocker cushion material, and the tension of the supporting springs produce errors of less than 0.1 percent of total sample on any sieve and hence need not be rigidly controlled. It has been demonstrated that a speed of 115 taps/min is superior to the usual

speed of 150 taps/min. The reproducibility is the same at speeds of 80 to 165, as long as the speed is constant. However, a minimum amount is retained on the sieves at 115 taps/min, and apparently the best measure of particle diameter is obtained. The two types of Ro-Tap machines (one or two eccentrics) are equivalent at 115 taps/min, but they are not equivalent at 150 taps/min. The differences between sieving analyses obtained on different machines are probably due entirely to differences in speed.

The errors inherent in the sampling and sample dividing procedure are not considered in this paper. The characteristics of the material can, however, be the source of some errors in sieve analysis. The density of the material being sieved has apparently no effect on the sieving characteristics. Nearly all bone chars are hard enough so that negligible wear occurs during a sieve analysis. Moist bone char can be satisfactorily sieved provided it does not appear moist and that the variation of moisture with particle size is properly evaluated.

The errors introduced by variations in weight of sample can be tremendous if very small or very large samples are used. Samples of 50 to 500 g of bone char can be satisfactorily sieved. However, it is recommended that samples of 100 to 150 g be used. The shaking time should be adjusted in accordance with the weight and particle size distribution of the sample.

The over-all reproducibility of a sieve analysis carried out in accordance with the following recommendations will be about 0.1 percent of the weight of the total sample. This variation will appear on all sieves irrespective of the amount of material retained on each individual sieve.

VI. Recommendations for Sieve Analyses of Bone Char

1. All testing sieves should be tested for uniformity of openings.
2. The effective openings of all testing sieves should be measured by means of a calibrated sample of material such as glass spheres, and the effective opening should be used instead of the nominal opening. All sieve analyses may then be calculated to correspond to the nominal openings.
3. If a Ro-Tap machine of the present design is used, it should be operated at 115 taps/min of the knocker mechanism. The other variables concerned with the shaking procedure are of minor importance and need not be rigidly controlled.
4. The sample should be dried before sieve analysis, although 1 or 2 percent of moisture in bone char has negligible effect.
5. The weight of the sample of bone char should be between 100 and 150 g, and the shaking time should be adjusted accordingly.
6. Fractions should be weighed

to the nearest 0.1 g and recorded to the nearest 0.1 percent. For purposes of uniform comparison between laboratories it is recommended that cumulative percentage finer be used to express the results of sieve analyses.

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VIII. Appendix

An analysis of variance for the top sieve takes the following form (for terminology, see any standard text on the analysis of variance, for example [18]):

Analysis of variance including all four knockers

[The value of F for significance at the 5-percent level is about 10 and at the 1-percent level about 30.]

Source of variation	Degrees of freedom	Sum of squares	Variance	F value
Knockers	3	3.255	1.085	93.0
Machines	1	0.0625	0.0625	5.36
Chars	1	131.1	131.1	11,230
Interactions: Machines and chars	1	0.16	0.16	13.7
Machines and knockers	3	2.0625	.6875	58.8
Chars and knockers	3	0.0525	.0175	1.5
Triple interaction (error)	3	.035	.01167	-----
Total	15	136.73	-----	-----

Since it is noted that the absence of a knocker (condition knocker 4) is the greatest cause of the variation, the above analysis of variance is repeated omitting K_4 :

Analysis of variance omitting K_4 (no knocker)

[The value of F for significance at the 5-percent level is about 20 and at the 1-percent level about 100.]

Source of variation	Top sieve		Second sieve		Third sieve		Pan	
	Variance	F	Variance	F	Variance	F	Variance	F
Knockers	0.0258	1.49	0.01	3	0.2580	1.2	0.01	-----
Machines	.8533	49	.0208	6.2	.1633	5.3	.48	-----
Chars	98.06	5,600	6.02	1,800	6.45	209	.03	-----
Interactions:								
Machines and chars	0.120	6.8	0.0208	6.2	0.0533	1.8	.12	-----
Machines and knockers	.0308	1.8	.0133	4	.0108	2.8	.01	-----
Chars and knockers	.0583	1.5	.0133	4	.0358	1.2	0	-----
Triple interaction (error)	.0175	-----	.0033	-----	.0308	-----	0	-----

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Research Paper 2144

First Spectrum of Arsenic

By William F. Meggers, Allen G. Shenstone,¹ and Charlotte E. Moore

The spectrum emitted by neutral arsenic atoms was observed photographically in the infrared, visible, and ultraviolet, and new lines were discovered in each spectral region. Measured wavelengths and estimated relative intensities are given for 330 lines, ranging from 1407.34 to 11679.9 Å in wavelength and from 1 to 2000 in intensity. More than 74 percent of the total number and 97 percent of the total intensity of observed lines have been explained as combinations of 30 odd energy levels arising from $4s^2 4p^3$ and $4s^2 4p^2 np$ electron configurations and 58 even levels from $4s 4p^3$, $4s^2 4p^2 ns$, and $4s^2 4p^2 nd$. The average difference between observed and computed wave numbers is 0.14 cm^{-1} . Most of the observed levels have been assigned to doublet and quartet terms, and spectral series of the type $4s^2 4p^3 - 4s^2 4p^2 ns$ have been identified. Calculations based on these series yield an absolute value of 79165 cm^{-1} for the ground state $4s^2 4p^3 \ ^4S_{3/2}$ of neutral arsenic atoms, that is, an ionization potential of 9.81 ± 0.01 electron volts.

I. Introduction

In 1929 Meggers and deBruin [1]² published a paper on the arc spectrum of arsenic, based on measurements of 54 ultraviolet lines (1889.85 to 3119.60 Å) and 23 infrared lines (7410.07 to 10023.98 Å). At that time this spectrum had not been observed in the region of shorter waves, and the assumption that the Blochs [2] had observed As I lines in spark spectra in the extreme ultraviolet could not be verified. Although it was not possible then to determine absolute term values from spectral series, the ground state was recognized as $^4S^o$, and by comparison with analogous terms in the spectra of neighboring elements the absolute value of this ground state was tentatively estimated as 80693 cm^{-1} , which corresponded to an ionization potential of about 10 eV. This was 13 percent lower than the value 11.54 ± 0.5 eV derived in 1922 by Ruark,

et al. [3] from experiments on low-voltage arcs in arsenic vapor.

A paper on spectra of arsenic in the extreme ultraviolet, 2500 to 710 Å, by Queney [4] reported about 300 arsenic lines from electrodeless discharges but added nothing to the As I spectrum.

The shorter waves of this spectrum were first investigated by K. R. Rao [5], who burned metallic arsenic in an arc between carbon poles and photographed the spectrum with an evacuated spectrograph containing a 1 m-radius grating, giving a scale of 8.6 Å/mm. He measured 64 lines between 1995.45 and 1563.08 Å with estimated probable errors of ± 0.03 Å. Because no spectral series could be found, Rao adopted the absolute value $^4S^o = 93500 \text{ cm}^{-1}$ from the ionization potential 11.54 eV reported by Ruark, et al. [3].

In 1932 further investigations of the arc spectrum of arsenic were reported by A. S. Rao [6], who measured 160 ultraviolet As I lines (1995.45 to 1319.48 Å) on a hollow-cathode spectrogram having a scale of

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² Figures in brackets indicate the literature references at the end of this paper.