DETERMINATION OF PARTICLE SIZE DISTRIBUTION—APPARATUS AND TECHNIQUES FOR FLOUR MILL DUST

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VOL. LIII NO. 2 JANUARY 5, 1950

Entered at the post office in Minneapolis as semi-monthly second-class matter, Minneapolis, Minnesota. Accepted for mailing at special rate of postage provided for in Section 1103, Act of October 3, 1917, authorized July 12, 1918.
ACKNOWLEDGMENT

The author wishes to acknowledge the financial support of the Millers' National Federation and their guidance through their members on the Curriculum Advisory Committee of the Institute of Technology, University of Minnesota.

The author would also like to express his appreciation to Professor John M. MacKenzie of the Mechanical Engineering Department for the help and encouragement given in the performance and publication of this investigation and to the Engineering Experiment Station, University of Minnesota, for cooperation in furnishing facilities for this investigation.
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INTRODUCTION

The problem of determining the particle size distribution of various materials has been thoroughly covered in many aspects by previous investigators. Therefore, as it exists today, the problem is chiefly one of adapting existing methods to the measurement of a particular substance over a specified range by developing apparatus and modifying the technique to give the desired accuracy at a minimum investment of equipment and time. With the above factors in mind the Milling Engineering Division of the Department of Mechanical Engineering set out during the summer of 1948 to develop methods for measurement in the subsieve range as a preliminary step in the study of mill dusts. An extensive library search was made, first to obtain a complete bibliography on the general subject of particle size measurement and second to search out information applicable to the problem at hand. Although this study was made with reference to a particular problem, it is felt that the bibliography and the outline of the subject is of sufficient value to be published. Therefore, the bibliography and outline along with a subject classification are to be published in a separate circular.

THE PROBLEM

Since the smaller particles of mill dust are most difficult to collect, the study is devoted primarily to the development of methods that would be most suitable for the subsieve particle size range (1 to 37 microns). However, it has been recognized that there is still a need for a rapid method that would successfully cover the particle size range of flour, that is from 5 to 150 microns. Therefore, some work has been done to extend the range of methods that were found suitable for the subsieve range.

Mill dust and other mill products have certain characteristics which require careful consideration when selecting methods. Mill products have a low specific gravity, usually less than 1.5. They are easily reduced by attrition when preparing microscope slides or making a sieve analysis. Care must be taken to select suspending mediums that do not affect the particles either by changing their size or density. Particle shape varies between specimens, sometimes over the range of sizes, and particles tend to aggregate. Mill products have a wide range of particle size; flours, for example, may have particles ranging in size from 2 to 150 microns. All of these characteristics tend to impose certain limits on the application of the various methods.
The techniques and apparatus developed for any one class of material must compromise accuracy, speed, range and cost. For research work a high degree of accuracy is required, whereas a method used for production control may be able to sacrifice some accuracy to save time. Some methods do not have flexibility with regard to the range that may be covered. Other methods are suitable only for a certain range. For example, sieving is limited to the measurement of particles above 37 microns in size (12).

METHODS STUDIED BY OTHER INVESTIGATORS

Work (13) has described microscopic methods rather completely. He has outlined methods of measurement, computation and presentation of data. Though microscopic measurement involves many problems and is tedious, it is fundamental because it permits direct observation and measurement of the particles.

Hildebrand, Ferrari, Borchardt and Ankar (6) have applied microscopic measurement of particle size distribution to mill products. Their method of preparing a representative, well dispersed sample on the microscope slide has overcome a major difficulty.

Wichser (12) has developed a sieving technique for the size analysis of flour, but the method is limited to particle size ranges above 37 microns.

Methods involving the rate of sedimentation of the particles in a fluid have been developed by many investigators. A direct weighing sedimentation technique developed by Oden (10) makes use of a balance pan submerged in the particulate suspension. The time-weight data determined with this apparatus is then analyzed mathematically or graphically and Stokes' law applied to obtain the particle size distribution. Oden and other early investigators found that some of the particles passed around the pan, thus giving erratic results. Hildebrand and associates (6) solved this problem by using a pan larger in diameter than the settling column and placing it directly below the column. Although satisfactory accuracy was obtained, their apparatus was complex and rather difficult to clean.

While experimenting with the Oden type apparatus, Jacobsen and Sullivan (7) developed a modification of the direct weighing method which solved many of the problems encountered by previous investigators. The essential improvement that they made was to extend the edges of the weighing pan upward to within a millimeter of the surface of the suspension, thereby preventing the particles from passing around the pan. With relatively simple equipment they obtained good results on high density inorganic materials. The
further development of this apparatus for measurement of the particle size distribution of mill products is the main subject of this paper.

Turbidimetric determinations have also been applied to measurement of particle size. Kent-Jones (8) has described the use of a turbidimetric method for determining the size distribution of flours. Essentially this apparatus measures the projected area of the particles in suspension at a given level by measuring the intensity of a transmitted beam of light. By applying Stokes' law it is possible to obtain the approximate particle size distribution from this data. The accuracy of this method is limited, however, because light transmission is a function of mass and of specific surface. Further research and calibration may extend the application of this method and it may eventually become an important method for particle size measurements.

Wichser (12) and others have also used the Andreason sedimentation apparatus for particle size measurement of mill products. Accurate results can be obtained if temperature control and very low concentrations of solid material are used.

Continental millers have also used the Werner sedimentation apparatus (11) in which depth of the material deposited in the bottom of a sedimentation column is directly measured on a scale at given time intervals. Though the method is simple, accuracy is poor due to the large concentrations necessary to get a measurable height of material settled in the calibrated tube.

Experiments by Stairmand (11) and Wichser (12) have shown that elutriators, although useable for separating the sample into fractions, have some serious disadvantages. Stairmand's work with the Roller air analyzer, for instance, has shown that fractions separated are not sharply defined, that the upper limit for sizing is 80 microns, and that the time necessary for a complete analysis may be long.

This brief survey of methods employed by other investigators has been presented to show the need for further research correlating the results obtained by the various methods, and more specifically the need for a rapid simple method of measuring the size distribution of flour mill products over a range of 2 to 200 microns.

METHODS INVESTIGATED

After a study of the methods used by previous investigators, a microscopic and three sedimentation methods were selected
for further investigation. Microscopic measurement was selected primarily because it gives basic data on shape. The Andreason, Dotts, and Oden sedimentation methods were selected for simplicity of apparatus. The Oden direct weighing sedimentation method offered the greatest possibility for development of a rapid automatic or semi-automatic device.

Inclined Manometer Sedimentation Apparatus

Dotts (4) has devised a simple apparatus for determination of particle size distribution by observation of the pressure changes in a sedimentation column.

Satisfactory results have been reported for relatively high density materials. The sensitivity of the apparatus decreased rapidly, however, with a reduction in the density of the dust or its concentration in the suspending liquid. The low density of flour dust made concentration of at least 5 per cent by volume necessary in order to obtain measurable pressure changes. As will be shown later, about 2 per cent by volume is the highest concentration that can be used without serious interference between falling particles. Also the time required for the measurement of particles under 10 microns was excessive, being in the neighborhood of four hours. No data obtained with this apparatus has been included because it is believed that the results are too erratic.

Andreason Sedimentation Method

The Andreason sedimentation apparatus (2) consists of a glass cylinder about 25 cm high and 5 cm in diameter with a special withdrawal pipette attached to a ground glass stopper. The pipette is fitted with a three-way stopcock so that samples of the suspension of dust in liquid can be withdrawn periodically into an evaporating dish. The residue after evaporation is weighed and, by application of Stokes' law, the particle size distribution is determined.

Though the apparatus is simple, care is required to get reproducible results. It was found that at a concentration of 5 gms per 100 ml the curve was shifted so that the distribution appeared 25 per cent finer than at a concentration of 2 gms per 100 ml. Stairmand (11) has shown that concentrations as low as 0.2 gm per 100 ml are necessary to completely eliminate interference between falling particles. A further discussion of the effects of concentration will be made in connection with the direct weighing method. Temperature control of the suspension to within 0.2 F was found necessary for good results. The time necessary
Microscopic Determination of Particle Size Distribution

Measurement by microscope can be considered fundamental because it permits visual comparison of size and direct observation of particle shape. The latter is particularly important when measuring mill products because particles in different size ranges may have different shapes. Figures 2 and 3 show clearly that the large particles, 75 to 150 microns in size, are much more angular in

![Graph](http://via.placeholder.com/150)

**FIG. 1**

**PARTICLE SIZE BY SEDIMENTATION - ANDREASON RYE DUST**

for complete evaporation was also found to be a further disadvantage. However, if a good analytical balance and an oven are available, the Andreason method may be useful where low cost is of primary importance.

Figure 1 presents the particle size distribution curve of a rye flour dust determined by the Andreason apparatus at a concentration of 2 gm per 100 ml.
Figure 2. Microphotograph of a family pastry flour 335X.

Figure 3. Microphotograph of poor roll suction 335X.
shape than the round starch granules, 15 to 40 microns in size. As will be shown later, this variation in shape with size affects the size distribution as determined by microscope or sedimentation. Figure 4, a microphotograph of a rye dust, shows the predominance of starch granules in this relatively fine sample.

It might be noted at this point that the particles in the microphotograph are much closer together than they would be on a slide to be used for counting. The more dense dispersion was necessary to get a satisfactory photograph.

![Microphotograph of rye flour dust 480X.](image)

**Apparatus**

When measuring particles with a microscope, microprojection is almost a necessity. Measurement with a filar eyepiece or eyepiece graticules is extremely hard on the eyes and very slow. Microphotography is also ruled out by the large number of fields that it is necessary to count.

Figure 5 is a photograph of the microprojector setup used for most of this work. An ordinary microscope equipped with a substage condenser and a mechanical stage is used. The photograph shows a ten cent mirror mount above the occular to reflect the image.
onto the screen. Though fairly satisfactory, this was later replaced by a reflecting prism when it became available. The screen consists of a sheet of transparent coordinate paper, ruled in cm and mm, placed between two sheets of window glass in an ordinary picture frame. It was found that ruling of the screen into 4 cm squares further facilitated the counting procedure.

A 6 volt, 108 watt Cenco ribbon filament lamp gives sufficient light to enlarge the image about 500 diameters. Though an arc lamp would give a much brighter image and permit magnification of 1000 diameters, it is believed that tungsten filament lamps are less expensive and less troublesome to operate for long periods.

Since it was desired to have the apparatus portable and flexible, the various components are merely set on a laboratory table as shown. Calibration of the image on the screen is accomplished by calibrating the filar eyepiece with a stage micrometer and then using the filar to calibrate the image on the screen. This is much more convenient than using the stage micrometer directly.

Preparation of Microscope Slides

The preparation of microscope slides to be used for the determination of particle size distribution poses a number of problems. Since the actual weight of the sample on the slide may be only a fraction of a milligram, the problem of obtaining a representative sample is a formidable one.

The necessity of a good dispersion in which the large and small particles are discrete and evenly distributed over the slide has been shown by many investigators. However, many of the
dispersion methods developed by previous investigators are not applicable to mill products because of the wide range of particle sizes that may be encountered. Hildebrand, Ferrari, Borchardt and Anker (6) obtained satisfactory dispersions of flour by using a fine pointed glass rod to disperse a milligram or so of flour on a slide, with dilute collodion. This method is satisfactory for fairly large particles above 20 microns, but it was found that samples of dust with a mean size under 20 microns tended to aggregate as the material was stirred. Also, it was found that this method had one other disadvantage. The size of the sample placed on the slide was so small that difficulty was experienced in obtaining a representative dispersion.

To overcome some of these difficulties, another dispersion technique has been developed. The dispersion medium is prepared by dissolving half a ml of cellulose acetate (airplane dope) in 200 ml of amyl acetate. Then, approximately 50 mg of the dust is added to 10 ml of this solution contained in a dropping bottle. After shaking vigorously, a drop is quickly placed in the middle of a clean slide and allowed to evaporate without being disturbed. This technique permits a larger and more representative sample to be used and gives a better dispersion. However, as the drop spreads on the slide, it tends to carry some of the finer particles to the outside giving a non-uniform dispersion along a radius from the center of the drop. At first it was believed that this might cause a serious error in the count. Figure 6 shows, however, that counts on slides prepared by each method are not significantly different. For this test a very carefully prepared sample of a patent flour was used to obtain a representative sample for each slide.

Measurement and Counting Procedure

Work (13) has described a good counting technique for uniformly dispersed particles. Basically the procedure outlined is as follows:

Measurement of particles is started on one edge of the dispersion. As soon as one field is measured, the slide is moved to bring the adjacent field into view. This process is continued until the dispersion has been crossed. If one traverse gives an insufficient number of particles, the slide is then moved just enough to bring a new field into view, as shown in Figure 7, and another row of fields counted. This procedure is continued until about 600 particles have been counted.

Figure 8 shows a sample data sheet. As can be seen from the first count column, a large number of small particles must be
FIG. 6
EFFECT OF TYPE OF MOUNT
MICROSCOPIC DETERMINATION OF PARTICLE SIZE OF REG. PAT. FLOUR

PATH OF OBSERVATION ON SLIDE
WHEN MEASURING PARTICLES
WITH MICROSCOPE

FIG. 7
METHOD OF MEASURING PARTICLES
ON MICROSCOPE SCREEN
<table>
<thead>
<tr>
<th>Size Range</th>
<th>1st Count</th>
<th>2nd Count</th>
<th>Corr. Count</th>
<th>% Size</th>
<th>Mean Size (5)x(6)</th>
<th>Wt. Size</th>
<th>% Finer Than Mean Size</th>
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<tr>
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<td>443</td>
<td>x3.26=1445</td>
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<td>500</td>
<td>360</td>
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<td>1.79 7.94</td>
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<td>823</td>
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<td>5.89 16.5</td>
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<td>5700000</td>
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</table>

23 = (No. above 40)  77 = (No. above 40)

\[
\frac{77}{23} = 3.26
\]

Figure 8. Data sheet for microscopic analysis.
counted to measure sufficient particles in the larger size ranges. Experience has indicated that at least 40 particles in the upper two-thirds of the size range of the sample must be counted for satisfactory accuracy. To increase the accuracy and reduce the labor involved, a multiple count method suggested by Work (13) is used. The procedure is to count particles in all size ranges on the first count until perhaps a total of 600 particles has been measured. Then, a second count is made, counting only particles above a certain range; in this case, 40 microns. Then, these two counts are added together, the sum being placed in column 4. Next the ratio of the sum of particles above 40 microns in column 4 is multiplied by the number of particles in each size range below 40 microns in column 1, to give the rest of the corrected count in column 4. Greater accuracy could be obtained by making further subdivisions and making more counts, but two or three stages have been found to be sufficient for most samples.

The method of measurement of the particles on the screen is important. Since only two dimensions are visible, it must be assumed that the third dimension is the average of the other two. Actually it is known that this is not true because the particles will tend to arrange themselves with their least dimension perpendicular to the slide. It will be shown later that this error will be relatively constant for a given material and is, therefore, of interest only when comparing distributions determined by different methods. Of the several different ways it is possible to measure the particle, Faires (5) gives measurement along horizontal lines dividing the particle into equal parts as a practical method, Figure 7. It is rapid and gives results fairly close to the average dimension. 

Expression of Results

Usually it is desirable to express the particle size distribution in cumulative percentages by weight finer than size, Figure 9. The problem is to translate the percentage of total count by size range to percent finer than a given size by weight. The first step is to determine a particle size representative of the average weight of the range. Since the mass of a particle is proportional to the cube of its size, the cube mean size is selected as representative of the average weight. It will be noted from column 10, of the data sheet, Figure 8, that the cube mean size is not the average size in the range. Thus, the particle diameter corresponding to the average weight of a 10 and a 20 micron particle is 16.5 microns and not 15. In practice this cube mean diameter is calculated as follows:
RESULTS

Cube of 10 = 1000
Cube of 20 = 8000
Sum = 9000
Average = 4500

Cube mean size $= \sqrt[3]{4500} = 16.5$

The second step in the translation of results, from percent of total count in each size range to percent finer than a given size by weight, is multiplication of the percent by size range (column 5) by the cube of the cube mean size (column 6).

Column 8 gives the percentage by weight in each size range, column 9 gives the cumulative percentages finer than the size in column 10.

It should be mentioned that equal size intervals were used in this work because unequal size intervals somewhat complicate the calculations.\(^1\)

Results

Figures 6, 9, 10, and 11 show cumulative distribution curves obtained by microscopic measurement. Figure 6 was made from a very carefully prepared sample and shows that results can be reproduced in successive examinations if a representative sample can be prepared. The other curves, however, show that random samples taken from a several hundred gram sample can give varying distributions. Thus, it is necessary to take great care in selecting the sample, and also necessary to obtain the average of several runs for dependable results.

Determination of Particle Size Distribution by Direct Weight Sedimentation Apparatus

Application of Stokes' Law

The direct weight sedimentation method originally worked out by Oden (10) is similar to other sedimentation methods in that

FIG. 9
MICROSCOPIC DETERMINATION OF PARTICLE SIZE
MULTISTAGE COUNT - RYE FLOUR DUST

FIG. 10
MICROSCOPIC DETERMINATION OF PARTICLE SIZE
MULTISTAGE COUNT - FAMILY PASTRY FLOUR
APPLICATION OF STOKES' LAW

it uses Stokes' law to relate the velocity of the settling particles to their diameter. In symbol form, Stokes' law is:

\[ V_m = K_s \left( \frac{\rho - \rho_0}{\rho_0} \right) D^2 \frac{1}{\nu} \]  

(1)

where \( V_m \) = terminal velocity of the particle falling freely in a fluid - cm/sec.

\( D \) = diameter of the particle - cm

\( \nu \) = kinematic viscosity of the fluid - Stokes'

\( \rho \) = density of the particle - gm/cc

\( \rho_0 \) = density of the fluid - gm/cc

\( K_s \) = a constant depending on the acceleration of gravity and the shape of the particle

\( \mu \) = viscosity of fluid - poise

Now: \( g = 980 \text{ cm/sec}^2 \)

\[ \nu = \frac{\mu}{\rho_0} \]  

(2)

Also from experiment we know that for a sphere falling in a fluid \( K_s = 54.5 \). If we now rearrange (1), solve for the diameter and introduce the constants, Stokes' law becomes:

\[ d^2 = \frac{18 \times 10^8 \mu h}{(\rho - \rho_0) g t} (S_K)^2 \]  

(3)

where \( d \) = diameter of particle - microns

\( h \) = height of fall - cm

\( t \) = time to fall a distance \( h \) - sec

\( S_K \) = correlation factor
Stokes' law, however, relates diameter and terminal velocity of a particle only for streamline flow. Allen (1) has given the following formula for calculating the largest particle which will fall according to Stokes' law under given conditions.

\[
D = 10^4 \sqrt[3]{\frac{36 \mu^2}{g \rho(\rho - \rho_0)}}
\]

Though there are more complete equations that will give the velocity of a falling particle for all types of flow, these equations are much too complicated for general application. For this reason, practically all methods of determining particle size by sedimentation choose liquids of such density and specific gravity that the limiting diameter exceeds that of the largest particle to be measured.

Table I gives the approximate density, viscosity and the diameter corresponding to the upper limit of Stokes' law, for several fluids used in this work. In general, the limit can be raised by increasing viscosity and density.

# TABLE I

Limiting Stokes' Diameter and Physical Constants for Various Liquids Used

<table>
<thead>
<tr>
<th>Liquid</th>
<th>( \rho ) 72°F</th>
<th>( \mu ) 72°F</th>
<th>Limiting ( d ) - microns*</th>
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</thead>
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<tr>
<td>Benzene</td>
<td>.8775</td>
<td>.00638</td>
<td>1.44</td>
</tr>
<tr>
<td>Gasoline</td>
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<td>.0055</td>
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<tr>
<td>Kerosene</td>
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<td>.01856</td>
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</tr>
<tr>
<td>1 part SAE - 10 oil to</td>
<td></td>
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<tr>
<td>1 part Kerosene</td>
<td>.8464</td>
<td>.0812</td>
<td>784</td>
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</table>

*Assume a dust density of 1.44.

---

**Figure 12.** Analytical balance as used for determination of particle size distribution by sedimentation.
FIG. 13
DIRECT-WEIGHT-SEDIMENTATION APPARATUS
Andreason (2) found that Stokes' law could be applied to angular or cubical particles of the same weight as spherical particles. By calculating the particle size as the edge length of a cube of the same volume as a sphere of diameter d, his particle size conformed to the results of sieve analysis. This shape factor \( S_k \) is found to be equal to 1.612.

All sedimentation results presented in this paper are calculated using \( S_k = 1.612 \), unless otherwise noted.

**Apparatus**

Briefly, the apparatus consists of a thin walled metal cylinder in the form of a cup 10.3 cm in height and 3.5 cm in diameter, suspended from a beam balance so that the upper lip is not more than 2 mm below the surface of the suspension. The suspension may be contained in a Dewar vacuum flask or a constant temperature bath. An analytical balance may be used, Figure 12, although the simple beam balance shown in Figures 13, 14, and 15 is much more convenient to operate, since no small weights need be handled. Some of the early runs were made with a constant temperature bath that would maintain the temperature within 0.1 F. However, it was found that an ordinary Dewar vacuum flask will maintain the temperature within 0.2 F under ordinary conditions.

If liquids that do not have known specific gravity and viscosity constants are used, it will be necessary to have a viscosimeter of the Ubeholdt or similar type. Density of the dust and of the liquid can be obtained in the usual manner with a 100 ml pycnometer bottle.

**Procedure**

**Preparation of the suspension**—The liquid selected for the suspending medium must meet certain requirements. Viscosity and specific gravity should be such that Stokes' law applies. The liquid must not affect the particles in any way. A non-volatile liquid is preferred because evaporation from the surface will cause convection currents in the suspension.

Benzene was used for measuring distribution in size ranges below 50 microns because its viscosity and density remain constant from one batch to another. Kerosene was used successfully for particles up to 150 microns, and a half-and-half mixture of kerosene and lubricating oil for suspensions containing particles larger than 150 microns.
Figure 14. Complete sedimentation apparatus for particle size measurement by direct weight method.

Figure 15. Direct weight sedimentation apparatus with flask removed showing cylinder.
SEDIMENTATION ANALYSIS

Sample 4  Run 2  From  
Date May 6, 1949  Observer Legg  

CALCULATION OF DENSITIES  Temp.  79.5°F  

(1) Wt. of bottle  = 8.4178 gms.  
(2) Wt. of bottle and liquid  = 16.2706 gms.  
(3) Volume of bottle  = 9,8000 cc  
(4) Wt. of liquid (2) - (1)  = 7.8528 gms.  
(5) Density of liquid \( \frac{(2) - (1)}{(3)} \)  = 0.8025 gms./cc  
(6) Wt. of bottle and powder  = 11.7869 gms.  
(7) Wt. of powder (6) - (1)  = 3.3691 gms.  
(8) Wt. of bottle, powder and liquid  = 17.7656 gms.  
(9) Density of powder \( \frac{(7) \times (5)}{(2) + (7) - (8)} = \frac{2.70}{1.874} = 1.439 \text{ gms./cc} \)  

STOKES' DIAMETER  

Suspending liquid kerosene  Viscosity .0176 poise  
Height of fall 10.45 cm.  Shape factor 1.612  

\[ D^2 = \frac{18 \times 10^8 n h}{(d - d_0) g t} \quad S_k = \frac{18 \times 10^8 \times 10.45 \times .0176}{(1.439 - .8025) 980.6 x t} x \left( \frac{1.612}{60} \right)^2 = 22900 \]  

MOISTURE CONTENT  

(1) Tare wt. of dish  = 27.2956 gms.  
(2) Dish and sample  = 29.3940 gms.  
(3) Dry sample and dish  = 29.1988 gms.  
(4) Per cent moisture \( \frac{(2) - (3)}{(3) - (1)} = \frac{.1952}{1.9032} = 10.30 \text{ per cent} \)  

Figure 16. Miscellaneous data sheet for sedimentation analysis.
After the suspending medium has been prepared, its viscosity and density should be determined. Then using the pycnometer bottle the density should be determined. Figure 16 illustrates a form for this determination that has been found convenient.

The concentration of particles in the suspending medium is important. Figure 17 shows a series of runs that was made on a carefully mixed sample of rye dust. This series of runs indicates that below a concentration of about 2 gm of dust per 100 ml of suspending medium, concentration has little effect on the results. For this reason, a concentration of 2 gm per 100 ml has been chosen for all of the work reported in this paper. Stairmand (11) indicates that free falling conditions are not approached until the concentration is less than 0.2 per cent by volume, but since these very low concentrations decrease the accuracy it is believed that nothing much is gained by using concentrations below about 2 per cent.

**FIG. 17**

**PARTICLE SIZE BY SEDIMENTATION**

**DIRECT WEIGHT METHOD - RYE FLOUR DUST**

**EFFECT OF PARTICLE CONCENTRATION**

Preparation of sample—When the apparatus is first set up, the volume of sample necessary to bring the liquid level in the flask 1 or 2 mm above the lip of the cylinder was determined. Also, the approximate weight on the balancing pan, with a blank
sample in the flask, was determined. Next, the weight of dust required for the desired concentration was placed in the predetermined volume of liquid and stirred from five to ten minutes with an electric stirrer. With the liquids used, no trouble was experienced in obtaining a thorough dispersion.

**Time-weight data**—While the sample was being stirred, the apparatus was checked, the thermometer put in place and the data sheets prepared. Then the dispersion was poured from the beaker into the flask, meanwhile continuously stirring the suspension in the beaker to prevent settling. As soon as all of the suspension was in the flask, the stop watch was started and a weight determined as rapidly as possible by one of two methods.

When using the analytical balance, it was found best to preset the weights and observe the times of balance but with the beam balance, Figure 13, it was found best to observe the weight at specified times. The time interval may be varied from 20 seconds at the beginning to 20 minutes at the end of 2 hours. It is important, however, to obtain as many readings during the first several minutes as is possible. Usually time-weight data was taken until the weight remained constant for two readings, but on some runs where the percentage of fines was unimportant, the run was terminated earlier. The sample data sheet, Figure 18, shows part of the data taken for run 1 of Figure 22.

**Calculation of results**—The first step in the calculation of the results was the plotting of the time-weight data as shown in Figure 19. The use of two time scales was found to increase accuracy and to reduce the size of the graph paper necessary. Earlier in this discussion it was mentioned that it is important to obtain several weights as soon as possible after the start of the run, because the first several points should form a line of constant slope until all of the largest size particles have reached the bottom of the can. Therefore, the straight line through the first several points was extrapolated to zero time in order to determine the weight at zero watch time. If it were obvious that the points did not define a straight line, then the run should be rejected. The final weight was determined by the horizontal portion of the curve or by the weight when the run was discontinued. The difference between the extrapolated weight at zero time and the final weight was then taken as the total weight settled.

Next, tangents to the curve at various times were drawn. Then the percentage of the total weight of particles settling out between any two given times is the ratio of the distance between
**APPARATUS AND TECHNIQUES FOR FLOUR MILL DUST**

## SEDIMENTATION ANALYSIS

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<th>Run</th>
<th>From</th>
<th>Date</th>
<th>Observer</th>
<th>Remarks</th>
<th>Weight Settled</th>
<th>Temp. - °F</th>
<th>Correspond Size - U</th>
<th>Per Cent Finer</th>
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<td>1</td>
<td>May 6, 1949</td>
<td>Legg</td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td>1</td>
<td>Poor Roll Suction</td>
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<table>
<thead>
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Figure 18. Data sheet for sedimentation analysis.
the intercepts of the two tangents on the weight scale to the total change in weight. By calculating the size of particle which will have completely fallen out at the given times, the percentage by weight in the given size interval was determined. However, it is more desirable to express the results in cumulative percentages finer than a given size. Referring to Figure 19 and the data sheet, Figure 18, the following calculation will serve to show how the percentage finer than a given size is calculated.

**Problem:** To find the percentage of particles in the sample finer than the Stokes' diameter corresponding to a time of 3 minutes.

1. Draw a tangent to the weight-time curve at 3 minutes intercepting the weight axis. (point C)

2. Calculate the percentage of particles finer than size.

\[
\% = \frac{AC}{AB} \times 100 = \frac{8.6}{13.6} \times 100 = 63.2\%
\]

where \( AC \) = distance between intercept at 3 minutes and final weight

\( AB \) = total weight settled out.

3. Calculate the Stokes' diameter corresponding to the time of 3 minutes.

\[
d = \sqrt{\frac{18 \times 10^8 \mu h}{(\rho - \rho_0) g t \times 60}}
\]

where:

\( \mu = .0176 \) poise

\( h = 10.45 \) cm

\( \rho_0 = .8025 \) gm/cc (density of liquid)

\( \rho = 1.439 \) gm/cc (density of dust)

\( S_K = 1.612 \) (shape factor)

\( g = 980.6 \)

\( t = 3 \) minutes
WEIGHT VS TIME PLOT

PARTICLE SIZE BY SEDIMENTATION — DIRECT WEIGHT METHOD
POOR ROLL SUCTION — RUN 1
CONCENTRATION: 2 G/100 ML. KEROSENE

FIG. 19
FIG. 20
PARTICLE SIZE BY SEDIMENTATION
DIRECT WEIGHT METHOD - RYE FLOUR DUST

FIG. 21 - PARTICLE SIZE BY SEDIMENTATION
DIRECT WEIGHT METHOD - FAMILY PASTRY FLOUR
d = diameter of particle - microns

\[ d = \sqrt{\frac{18 \times 10^8 \times 0.0176 \times 10.45}{(1.439 - 0.8025) \times 980.6 \times 60 \times t} \times 1.612} \]

\[ = \sqrt{\frac{23000}{t}} = \sqrt{\frac{23000}{3}} = 87.5 \text{ microns} \]

Steps 1, 2, and 3 above, were repeated for each time at which it was desired to obtain a size and percentage; the resulting values were plotted to give the complete particle size distribution curve as shown in Figure 22.

Markley (9) gives an analytical method for obtaining the distribution curve from the time-weight data but it is believed that the graphical method is more accurate and rapid.

FIG. 22
PARTICLE SIZE BY SEDIMENTATION
DIRECT WEIGHT METHOD - POOR ROLL SUCTION
Results

Figures 20, 21, and 22 show the results of a number of runs on different mill products. It is to be noted that the largest deviations are at the coarse end of the curve. This is due to the close spacing of points in the initial straight portion of the time-weight curve. Increasing the viscosity of the suspending medium prolongs the period during which the largest particles are settling permitting the wider spacing of points in this segment of the time-weight curve and hence more accurate plotting.

Figure 23 is a comparison of the average of the microscopic and sedimentation analysis made on the rye dust. In this figure the sedimentation results have been plotted both with and without the shape factor of 1.612. Figures 24 and 25 also show a comparison of microscope and sedimentation results on a flour and a poor roll suction sample. A comparison of these curves shows that the microscope results do not correspond to the sedimentation results even with the factor, $S_K$. Further study of these and other sets of data indicate that there is a different shape factor for each size range. The factor for any given size range, however, appears to be common for all mill products covered by this study.
FIG. 24
AVERAGES OF SEDIMENTATION & MICROSCOPIC DETERMINATION
OF PARTICLE SIZE – FAMILY PASTRY FLOUR

FIG. 25
AVERAGES OF SEDIMENTATION & MICROSCOPIC DETERMINATIONS
OF PARTICLE SIZE – POOR ROLL SUCTION
The Andreason sedimentation technique has been considered as a standard by many investigators. A comparison of results on the rye dust as determined by the direct weight and Andreason methods is presented in Figure 26. Agreement is very good in the middle range of the distribution, but deviates at the end points.

A number of conclusions can be drawn from Figure 27. The fact is, that $S_K$ is greater than 1.612 above 30 microns and nearly constant. The microscope measurement is high due to particles having their least dimension perpendicular. We might assume that density and shape of the particles are nearly constant above that size. However, below about 30 microns the particles are predominantly spherical in shape and of higher density thus reducing $S_K$. It must be understood that this is a coefficient that accounts for several variables such as density variations with particle size, changes in shape, and the tendency for particles to lie flat on a microscope slide. Thus it would be possible to correct observed sedimentation results from the $S_K$ values for each size range as selected from Figure 27 giving very close agreement with microscopic observations.

Other Observations

Following are some useful observations made while developing the sedimentation apparatus:

1. The lip of the cup must be no more than 2 mm below the surface of the suspension for consistent results.

2. A concentration of no more than 2 gms of particulate material per 100 mls of liquid should be used. In any case the concentration should be kept constant to make runs comparative.

3. Vertical oscillations of the cylinder must be kept as low as possible, preferably less than 0.5 mm.

4. When using a volatile suspending medium a fairly tight cover should be fitted on the flask to prevent evaporation. Evaporation is serious because the cooling at the surface causes convection currents to form in the suspension.
APPARATUS AND TECHNIQUES FOR FLOUR MILL DUST

FIG. 26
ANDREASON VS. DIRECT WEIGHT SEDIMENTATION
RYE DUST

FIG. 27
RELATION BETWEEN STOKES' DIAMETER
AND MICROSCOPIC DIAMETER
MICROSCOPE DIA. = $S_k$ STOKES' DIA.
CONCLUSIONS

5. Temperature during a run should not vary more than 0.2 F. If the temperature of the suspension is at room temperature before it is put in the flask, this is easily maintained.

6. Graphical differentiation of the time-weight curve should be performed carefully, but errors tend to average out except at the end points.

Conclusions

The main purpose of this investigation was to select and correlate methods for determining subsieve particle size distribution that could be adapted to the study of mill dusts. Methods were desired which would be rapid, accurate, adapted to a wide range of measurement, reasonably simple to operate and relatively inexpensive. The purpose of this preliminary investigation was not to study dusts but to develop the methods of study. The achievement of this purpose is reflected in the following conclusions:

1. Two methods of measurement suitable for the subsieve range have been selected, namely direct measurement by microprojector and indirect measurement by means of the direct-weight sedimentation apparatus.

2. Satisfactory procedures for sample selection, sample dispersion, counting, and expression of results have been developed for the microprojection method.

3. Satisfactory apparatus has been developed, factors relating to its operation have been evaluated, and procedures for operation have been established for the direct-weight sedimentation.

4. Microprojection and sedimentation have been compared and the shape factor $S_K$ has been shown to vary with particle size.
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