AN INVESTIGATION OF SOME OF THE LAWS GOVERNING THE GRINDING OF MATERIALS IN BALL AND TUBE MILLS

WITH SPECIAL REFERENCE TO THE SIZE DISTRIBUTION OF THE PARTICLES IN THE PRODUCT.

Thesis

by

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SUMMARY.

INTRODUCTION.

1. General Statement of the Problem. The purpose of this investigation was to determine the effects of some of the factors involved in the grinding of dry materials in laboratory size ball mills. The major part of the work has been directed towards ascertaining the effects of variations in,

(1) Size of the material fed to the mill,
(2) Size of the balls used in the mill,
(3) Ratio of the amount of charge to the amount of balls in the mill,
(4) Time of grinding,

upon the size distribution of the resulting product and upon the amount of new surface produced for equal amounts (i.e. times) of grinding.

2. Defects of Old Experimentation. This problem has been attacked several times before. Practically all of the old investigations, however, have been handicapped by lack of information concerning the size distribution of the fine part of the product, i.e., that which passed through the finest reliable screens. This handicap has been very serious, as can be appreciated when it is realized that from 10 to 95 percent of the material involved lies in this class.

3. Defects of Recent Experimentation. The recent experimenters have tried to overcome this difficulty in many ways. All of the accounts of their work that could be obtained show that their methods for the determination of this size distribution

of fine particles has been such a laborious process that the experimenters have been limited to the study of comparatively few samples from a correspondingly few runs in their experimental mills.

This has made it impossible to cover very wide variations of the different factors involved in the grinding operation; or to allow of many check runs, either of mill operation, or of size distribution of the product.

4. Scope of the Present Investigation. The present investigation has endeavored to cover a wider field in a more intensive manner, in an attempt to reach conclusions that would be more universal in character than have hitherto been possible.

5. Method of Attack on the Previous Defects. When the problem was first considered it was felt that the possibility of success depended upon the development of a method for determining the size distribution of the fine material quickly and accurately. Therefore the work was divided into two parts; first, the development of a method and apparatus for size distribution analysis; second, a study of some of the phenomena of grinding in a laboratory mill, carried on with the aid of this apparatus.

6. Summary of Results.

a. Size Distribution Apparatus. An instrument, called the Microneter, has been developed by means of which it is possible to make routine analyses of the size distribution of particles ranging from 8 to 74 microns in diameter. The lower limit of this range can be shifted downward to 2 to 3 microns if desirable, at the sacrifice of speed. For routine analysis work with a lower limit of 8 microns two men and a helper have completed 30 analyses a day, continuously.

b. Laboratory Mill Studies. A large number of runs have been made in the laboratory jar mill. The major results obtained to date are illustrated most clearly by two series, Series E and No. 2 Time Runs. These two groups represent about three hundred separate runs, exclusive of any checks. The principal results are summarized below,

(1) Rittenger's Law, that equal expenditures of energy produce equal increments of surface, holds over wide limits for given conditions of ball size and initial charge particle size.

(2) The grinding efficiency is not affected by variations in the amount of charge present in the mill if the ball load is kept constant. However, the capacity is slightly decreased if the charge is larger than that required to fill the voids between the balls.

(3) The size distribution of particles in that part of the product passing the 200 mesh sieve depends upon the ratio of the initial particle diameter to the ball size. The larger this ratio the larger is the amount of very fine (-10 micron) material produced. The smaller this ratio the more uniform is the size gradation.

(4). The grinding efficiency depends upon the ratio of $\dfrac{\text{particle diameter}}{\text{ball diameter}^3}$. There is an optimum value for this ratio and deviations in either direction cause losses in efficiency.

(5) A tentative theory is offered that the amount of energy required for grinding depends upon the modulus of resilience of thematerial and the size reduction desired.

PART I.

THE DEVELOPMENT OF THE MICRONETER.

APPARATUS PREVIOUSLY EMPLOYED FOR DETERMINATION OF SIZE DISTRIBUTION.

7. Field of Application of Sieves to Determine Size Distribution. As has been stated in the introduction, (par.2) the need for information concerning the size distribution of the fine portion of the material worked with is very pressing. A study of a grinding operation inherently involves a comparison of the size distribution of the particles of a material before and after grinding to determine the effect of the grinding process. The size distribution has usually been obtained by means of sieves or screens. It is generally acknowledged that the 200 mesh sieve is the finest that it is feasible to use for routine analysis. Some of the studies which have been made in the course of the present program have shown that even this sieve, and in fact several larger sized sieves, give results that contain much greater possible errors than have been commonly recognized. However, consider the situation when it is accepted that screens offer a satisfactory method of determining the size distribution of material down to the size that remains on the 200 mesh sieve.

8. Inadequacy of Sieves for the Determination of the Entire Size Distribution. In the manufacture of cement it is very common to employ ball mills and tube mills; the former to reduce the material from a maximum size of 2 inches to particles of about minus 20 mesh; the latter to take this minus 20 mesh

material and reduce it to a product of which 75 to 95 percent will pass the 200 mesh sieve. This does not tell the complete story for often the feed to the ball mill contains 10 percent of material which passes the 200 mesh sieve and the product from the ball mill (i.e.,the tube mill feed) generally contains from 20 to 35 percent of this sized material. It is therefore rather obvious that an investigation into the action of ball and tube mill grinding would be severely handicapped without knowledge of the size distribution of such large fractions of the material under consideration.

9. Review of Other Methods Used to Determine the Size Distribution of Finely Divided Material.

Because of the great need for this information there have been many attempts made recently to develop some means of obtaining it. Numerous ways were brought forth in an attempt to measure some function which depends on the fineness of the material. For example, the ratio of the apparent density(1) of the dry material to its actual density has been suggested as a measure of its fineness, likewise, its rate of solution(2),(3),(4) in a given solvent, under standardized conditions, or its light dispersing power(5),(6) in given concentrations. However, all these methods are simply trying to get one figure which represents the average particle diameter or possibly the total surface of the sample, and do not pretend to give information about the size distribution of the particles. Therefore this type of attack was rejected because it is not designed to give the kind of data required by the investigation under consideration,however valuable such

(1) *References, p. 122*

information may be for other purposes.

The work that has had for its object the actual determination of the size distribution of the particles has, in general, proceeded along two main lines:

a. Microscopic Measurement Method. Of these the most direct method has been that of microscopic measurement.(1),(2),(7),(8) The following is a typical procedure:- a very small sample of the material is carefully spread on the microscope slide, protected with a cover glass, and then inspected either directly with a microscope or by projecting the field upon a screen where the particles are measured and counted. This may be done in several ways. If a high degree of accuracy is desired, all of the particles on the slide are measured and counted. If this is too tedious a ruled slide and screen are used and only particles falling in selected areas are counted; i.e., in a manner similar to a blood count.

b. Sedimentation Method. The other line of development grades the sample by taking advantage of the fact that the rate of fall of a particle in a fluid depends on its diameter. There have been many different methods based on this phenomena, which is expressed mathematically by Stokes' Law. For example, all elutriation apparatus(9),(10) and all sedimentation apparatus(11),(12),(13) employs this principle, regardless of whether the fluid is liquid or gas.

Elutriation methods separate their fractions by balancing the rate of all of a given size of a particle against the upward velocity of the fluid stream employed. Obviously all particles larger than the size balanced settle down against the current, while the smaller ones are carried up and collected in another plane. Sedimentation methods employ static bodies of fluid, and make their separation or measurment by using the different rates of fall of the various sized particles present.

10. Enumeration of the Desirable Properties of A Size Distribution Apparatus. At the beginning of the work, a rather careful study of the available methods of sub-sieve size determination was made, to ascertain whether it would be possible to obtain commercial apparatus to aid in this work. The following were set up to be the desirable features of such an apparatus:

(1) It should be accurate.
(2) It should be capable of making a large number of analyses per day.
(3) It should be preferably self recording.
(4) It should not change the size gradation of the sample.

11. Defects of Available Apparatus. It was soon found that there was practically no commercial apparatus available in this country. The same criteria were then applied to the various methods that had been used by other investigators. Microscopic measurement was ruled out because its extreme tediousness prevented it from being used for a large number of samples. Air elutriation was discarded because it was open to the grave suspicion of grinding the sample during the separation process itself, thus failing to fulfill criteria 1 and 4. Practically

all of the methods failed to meet requirements 2 and 3. However, three types of apparatus appeared to have considerable promise, and were therefore studied carefully. They are very similar, since all are sedimentation methods.

a. The Werner Sedimentation Apparatus.(13) This apparatus consists of a long narrow vertical tube which is filled with liquid. A small chamber, of the same diameter, fits on top and is separated from the main tube by a sliding gate. The bottom of the tube is carefully graduated. At the start of an analysis, the tube and chamber are filled with liquid and the gate closed. A given weight of the sample is placed in the chamber and thoroughly mixed with the liquid. The gate is suddenly opened and the sample allowed to settle. Readings are taken at given times of the amount of material collected at the bottom. If the relation between diameter and settling time is known, it is then possible to plot the size distribution curve from this data.

b. The Oden Sedimentation Apparatus.(11),(14) This method is illustrated in Fig.(1). The procedure is as follows:-

A sample of known weight is placed in cylinder A, which contains a definite

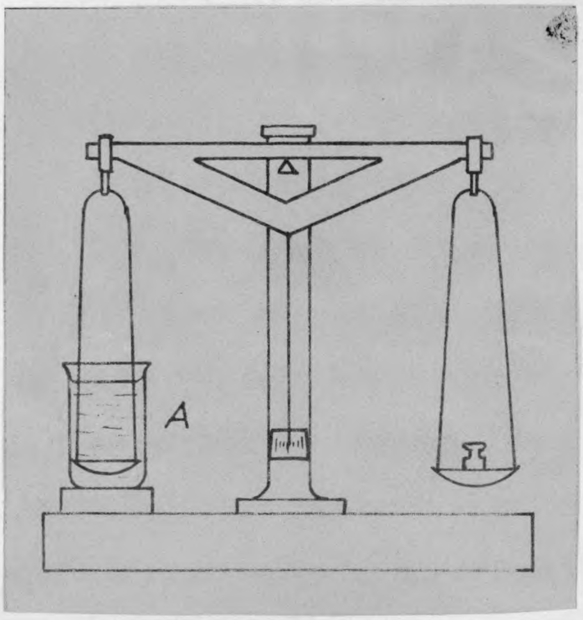


Fig. (1)

amount of liquid. This is vigorously stirred or shaken to insure a homogeneous mixture. The cylinder is then placed in an analytical balance, one pan of which is submerged nearly to the bottom of the cylinder. As the sample settles out, the increase of weight of the pan is recorded, together with the corresponding time intervals that have elapsed. Readings are taken until practically all of the sample has settled out. They are then corrected to eliminate a number of extraneous effects and plotted with the weight for ordinate time for abscissa.

c. <u>The Wiegner Sedimentation Apparatus.</u>(12),(15) The third method is very similar in principle. Fig. (2) shows a diagramatic sketch of this apparatus. A cylinder C of fairly large dimensions has a manometer tube M connected to it near the bottom. The cylinder is filled with liquid to some height H. The manometer tube of course fills to the same level. The valve V is then closed and a given weight of the material to be analyzed

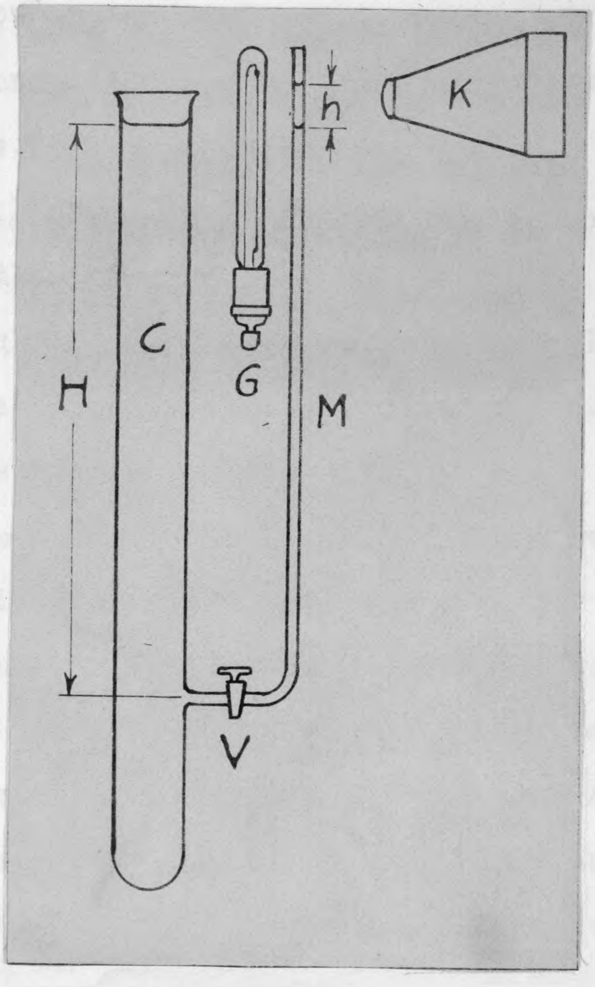


Fig. (2)

is added to C. This is thoroughly mixed, as before. When the mixing is complete, the valve V is opened. Since the average density of the liquid in C is now higher than that in M, due to the addition of the sample, the pressure in C at the point of connection is higher than in M and therefore the level in M rises a distance h. As the material in C settles below the manometer connection the level in M recedes and furnishes a measure of the amount of material in suspension in C. The height of the meniscus in M is recorded photographically by means of the camera K. The column is illuminated by the show case incandescent light G. The photographic plate is given a constant horizontal motion by a clock. This plate shows, upon development, a curve similar in shape and meaning to the one constructed from the data obtained by the Oden apparatus.

d. Discussion of Werner Apparatus. This apparatus has a number of good points, but at the same time a number of faults. Its chief advantage is that the data from it gives the actual distribution curve, while, as will be seen later, the curves plotted from the other methods are not of themselves distribution curves but only sources from which the distribution curves may be obtained. On the other hand it is very difficult to start all of the material from the top without causing "streaming" and eddy currents which would completely destroy the accuracy of the determination. Also, the measurement of the material settled out by observing its volume is open to question.

In common with the Oden apparatus, it demands frequent attention,

e. Comparison of the Oden and the Wiegner Methods. These two methods are seen to be quite similar in principle and result. However, the latter has several important advantages. First, it is self recording and hence does not require continuous attendance. Second, it has no foreign bodies suspended in the sedimentation chamber, and thereby avoids some troublesome corrections that must be made in the first method to get rid of changing buoyancy effects, etc. On the other hand, the Wiegner apparatus also has some defects. First, the movement of the meniscus is small, thus necessitating very accurate measurement of its change in level. Second, the height of the column in M is affected by the temperature of its own liquid as well as by the pressure from the adjacent chamber C. For example, the heat from the light G is found to be troublesome. Third, it is difficult to prevent the base of the manometer column from becoming contaminated with some of the sample from C.

THEORY UNDERLYING MICRONETER.

12. General Type of Apparatus Constructed for Present Investigation. It was finally decided to construct an apparatus on the principle of the Wiegner but differing in the methods of measuring and recording the pressure changes. An experimental model was built, consisting of a single unit of sedimentation chamber and recording mechanism. The results were encouraging enough to justify the design and construction of a

six unit apparatus for the use of the grinding studies. Before this apparatus is described, the principles underlying the operation of the machine itself and the method of handling the data to obtain from it the size distribution of the material will be discussed.

13. Stokes' Law. First, let us consider the general law governing the settling of particles in a fluid. Where they are small enough so that their motion through the fluid does not cause currents to be set up, viscous of stream line conditions hold and the equation expressing the relationships existing is called Stokes' Law and may be written as follows:-

$$v = \frac{2g(\sigma - \sigma_m)r^2}{9\eta} \qquad \text{I.}$$

Where

v = velocity of fall.
g = acceleration of gravity.
σ = density of the falling substance.
σ_m = density of the liquid.
η = the viscosity of the liquid.
r = radius of the particle.

Since, in the type of apparatus under construction, we are more interested in the time that the particle requires to fall a given height, the equation may be written,

$$T = \frac{H}{v} = \frac{9H\eta}{2g(\sigma - \sigma_m)r^2} \qquad \text{II.}$$

Where

T = time of fall
H = Height of fall

If the height, viscosity, and densities are held constant this becomes:

$$T = \frac{K}{r^2} \quad \text{or} \quad r = \frac{K'}{\sqrt{T}} \qquad \text{III.}$$

K and K' being constants.

K and K' being constants.

If a known weight of sample is allowed to settle a distance H, through a column of liquid, the relation between the weight of the material reaching the bottom and the time can be determined. By means of equation III, values of the radius r or diameter d can be substituted for the corresponding values of T. When the weight of material reaching the bottom in a given time is plotted against the corresponding diameter, (calculated from that time) a cumulative distribution curve of the usual type is obtained, Fig. (3).

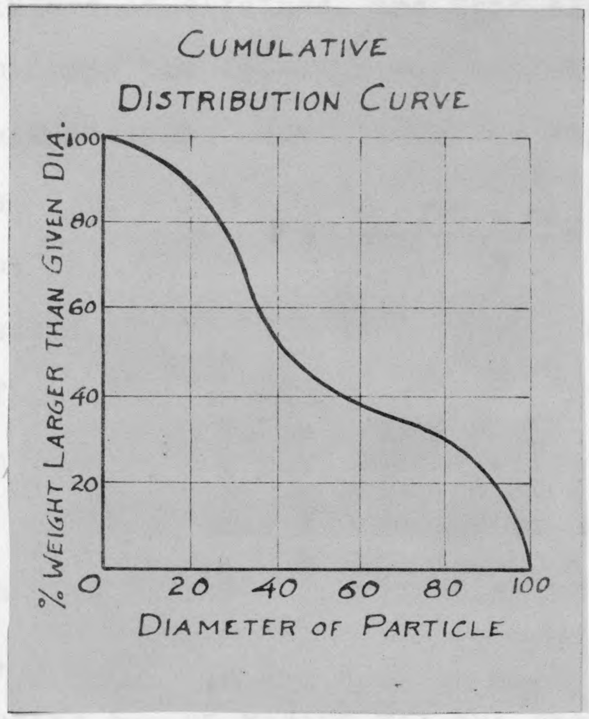


Fig. (3)

14. Practical Difficulties in the Use of this Method. There are practical difficulties in the way of carrying the procedure just outlined. The most serious one is encountered when an attempt is made to start the sample evenly from the top of the column. Since it is considerably more dense than the liquid, the layer of sample (whether dry or mixed with a small quantity of

liquid) tends to go 'en masse' to the bottom, forming numerous eddy currents in the process. This tendency is very pronounced, and, although it is possible to prevent it in a long, very narrow tube, there are other effects that make the procedure undesirable.

15. Conditions Encountered When Sample is Uniformly Mixed.

Of course the simplest method of starting a sedimentation is to stir the sample thoroughly into the liquid until a homogeneous mixture is obtained, and then allow it to settle. However, this entirely changes the relation between the amount of material settling out and the time of sedimentation. This is because, instead of settling out in layers of definite sized material, the sample now settles out particles of all sizes at once. Therefore the weight settles out is much greater at any given time than if all of the sample had been started from the top. Consider a hypothetical example:-

A sedimentation column of height H is available. Three samples of known uniform size are to be used; the first sample S_1 consists of particles all of which have a radius r; the second S_2, of radius r_2; and the third S_3, of radius r_3. All the constants of equations II and III are known. Substituting in the numerical values it is determined that the particles of S_1 will settle the distance of H in T_1 minutes, S_2 in T_2, S_3 in T_3.

First S_1 is placed on the sedimentation column, mixed thoroughly and allowed to settle. Since the particles are all of the same size they will all settle with the same velocity. As they are distributed uniformly throughout the column, the amount collected on the bottom will be a linear function of the time, being 0 percent at time 0, and 100 percent at the T_1, as shown in Fig (4) curve S_1.

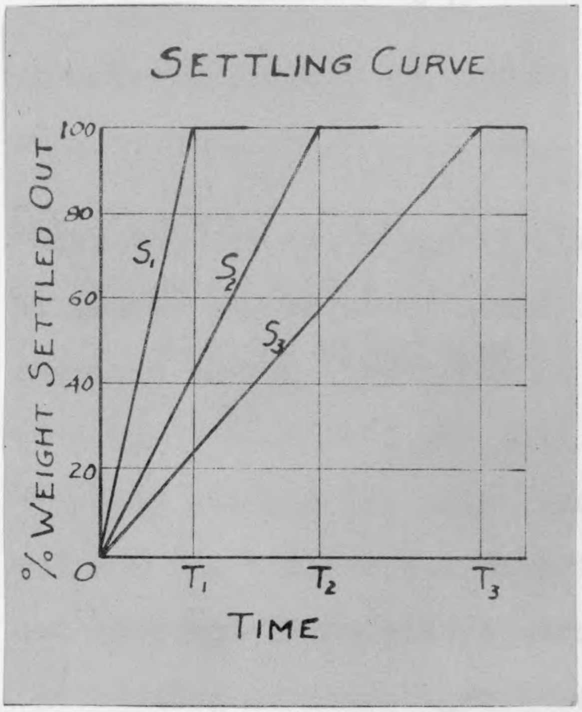


Fig. (4)

Likewise if sample S_2 is tested in the same manner the result is represented by curve S_2, and in the same way curve S_3 is obtained. Note that all of these relations are straight lines, because all the samples consisted of uniform sized particles, ~~which were particles~~ which were uniformly distributed in the sedimentation chamber.

Now, a sample consisting of one third S_1, one third S_2, and one third S_3 is placed on the sedimentation column. If S_1 were settling out alone it is obvious that the curve would be the one labeled S_1, in Fig.(5) since there is only 33 1/3 percent of S_1. Likewise S_2 and S_3 would be as shown, if either was the only one present.

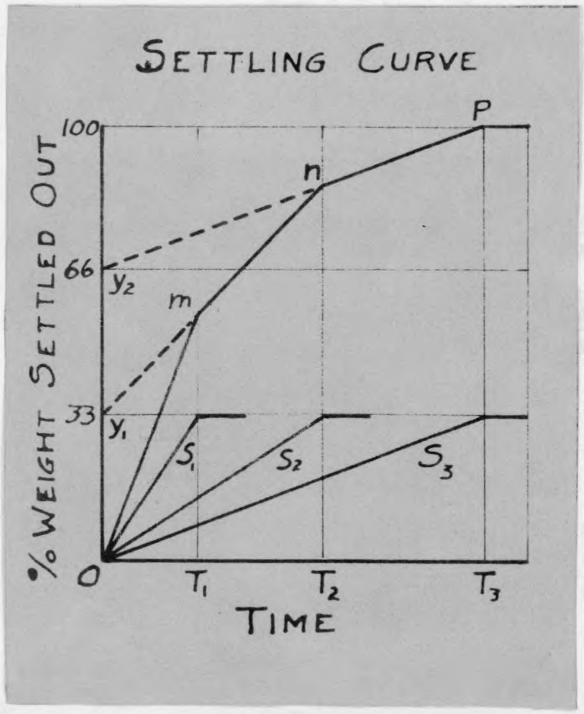


Fig. (5)

Since they are all settling together, in the time T_1, all of S_1 will be out, together with a percent S_2 and b percent of S_3; therefore the total percent out will be,

$$33\tfrac{1}{3} + a + b$$

$$\text{but } a = 33\tfrac{1}{3}\frac{T_1}{T_2} \qquad \text{and } b = 33\tfrac{1}{3}\frac{T_1}{T_3}$$

$$\text{or \% out} = 33\tfrac{1}{3}\left(1 + \frac{T_1}{T_2} + \frac{T_1}{T_3}\right) = m$$

Since the individual settling curves are straight lines, the composite curve will likewise be straight, going from o to m. At time T_2 all of S_1 and S_2 will be out and C $= 33\tfrac{1}{3}\frac{T_2}{T_3}$ of S_3

or $\% \text{ out} = 33\tfrac{1}{3}\left(1 + 1 + \frac{T_2}{T_3}\right) = n$

Similarly at T_3, all, the sample will have settles out, so point P = 100 percent. Thus it is seen that the settling curve is composed of the broken straight line o = m-n-p. It is interesting to investigate the point at which n-m produced would cross the y axis. This would be where,

$$y_1 = m - T_1\frac{n-m}{T_2 - T_1}$$

$$= 33\tfrac{1}{3}\left(1 + \frac{T_1}{T_2} + \frac{T_1}{T_3}\right) - T_1\frac{33\tfrac{1}{3}\left(2 + \frac{T_2}{T_3}\right) - 33\tfrac{1}{3}\left(1 + \frac{T_1}{T_2} + \frac{T_1}{T_3}\right)}{T_2 - T_1}$$

$$= \frac{33\tfrac{1}{3}}{T_2T_3}(T_2T_3 + T_1T_3 + T_1T_2) - \frac{33\tfrac{1}{3}}{T_2T_3}(T_1T_3 + T_1T_2)$$

$$\text{or} \quad y_1 = 33\tfrac{1}{3}$$

Likewise np produced is

$$y_2 = n - T_2\frac{p-n}{T_3 - T_2}$$

$$= 33\tfrac{1}{3}\left(2 + \frac{T_2}{T_3}\right) - T_2\frac{100 - 33\tfrac{1}{3}\left(2 + \frac{T_2}{T_3}\right)}{T_3\ T_2}$$

$$= \frac{33\tfrac{1}{3}}{T_3}(2T_3 + T_2) - \frac{33\tfrac{1}{3}T_2}{T_3}$$

$$\text{or} \quad y_2 = 66\tfrac{2}{3}$$

This is significant, since 33 1/3 is the percent of S_1 in the total sample and 66 2/3 is the sum of the percentages of S_1 and S_2.

16. Oden's Interpretation of Settling Curves. The above hypothetical example serves to illustrate the method used by Sven Oden(11) to interpret settling curves. He proved that the above relations hold in any suspension of different sized particles of the same substance where the sizes are such that they follow Stokes' Law. A general statement of the method is as follows:-

If the sedimentation curve is plotted with the time of settling as abscissa and the weight of material settled as ordinate, then the intercept on the y axis of a tangent to the curve at a point T will give the percent of the sample that has a diameter equal to or larger than that of a particle which would just settle a distance equal to the height of the sedimentation column in the time T.

17. Proof of the Tangent Method of Analysis. The work of Oden was not available, and no other source was found which gave the general proof of the above statement. Therefore the following derivation was developed:

Consider a sample composed of a large number of groups of different sized particles of a given substance. Let it be placed in a sedimentation chamber and data obtained to construct the usual sedimentation curve, Fig. (6).

Let

w_t = the weight of the fraction which has a diameter corresponding to that of a particle which just settles the height of the sedimentation column in the time t.

t = the time required for any fraction w_t to completely settle out (either if started all from the top or if uniformly mixed)

T = time of settling of one given fraction.

ΔT = the difference in time between T and next slowest settling fraction.

S = the slope of a tangent to the settling curve.

I = the y intercept of a tangent.

y = the general ordinate.

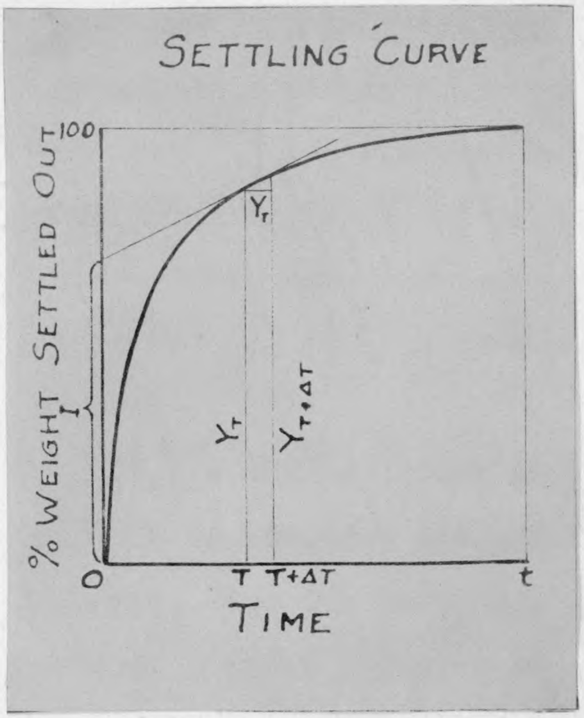


Fig. (6)

Construct a tangent at point T.

then:

$$S_T = \frac{y_{T+\Delta T} - y_T}{\Delta T}$$

AND

$$I_T = y_T - S_T \cdot T$$

BUT

$$y_T = \sum_0^T w_t + \sum_T^\infty w_t \frac{T}{t}$$

AND

$$y_{T+\Delta T} = \sum_0^{T+\Delta T} w_t + \sum_{T+\Delta T}^\infty w_t \frac{T+\Delta T}{t}$$

$$\therefore \quad S_T = \frac{\sum_0^{T+\Delta T} w_t - \sum_0^T w_t + \sum_{T+\Delta T}^\infty w_t \frac{T+\Delta T}{t} - \sum_T^\infty w_t \frac{\Delta T}{t}}{\Delta T}$$

BUT

$$\sum_{T+\Delta T}^{\infty} w_t \frac{T+\Delta T}{t} = \sum_{T+\Delta T}^{\infty} w_t \frac{T}{t} + \sum_{T+\Delta T}^{\infty} w_t \frac{\Delta T}{t}$$

ALSO

$$\sum_{T+\Delta T}^{\infty} w_t \frac{T}{t} - \sum_{T}^{\infty} w_t \frac{T}{t} = - w_{T+\Delta T} \cdot \frac{T}{T+\Delta T}$$

AND

$$\sum_{0}^{T+\Delta T} w_t - \sum_{0}^{T} w_t = w_{T+\Delta T}$$

$$w_{T+\Delta T} - w_{T+\Delta T} \frac{T}{T+\Delta T} = w_{T+\Delta T} \cdot \frac{T+\Delta T-T}{T+\Delta T} = w_{T+\Delta T} \cdot \frac{\Delta T}{T+\Delta T}$$

$$\therefore \quad S_T = \frac{w_{T+\Delta T} \cdot \frac{\Delta T}{T+\Delta T} + \sum_{T+\Delta T}^{\infty} w_t \frac{\Delta T}{t}}{\Delta T}$$

NOW

$$I_T = y_T - S_T \cdot T$$

$$= \sum_{0}^{T} w_t + \sum_{T}^{\infty} w_t \frac{T}{t} - \frac{T}{\Delta T}\left[w_{T+\Delta T} \cdot \frac{\Delta T}{T+\Delta T} + \sum_{T+\Delta T}^{\infty} w_t \frac{\Delta T}{t} \right]$$

$$= - w_{T+\Delta T} \cdot \frac{T}{T+\Delta T} + \sum_{0}^{T} w_t + \sum_{T}^{\infty} w_t \frac{T}{t} - \frac{T}{\Delta T} \sum_{T+\Delta T}^{\infty} w_t \frac{\Delta T}{t}$$

BUT

$$\frac{T}{\Delta T} \sum_{T+\Delta T}^{\infty} w_t \frac{\Delta T}{T} = - \sum_{T+\Delta T}^{\infty} w_t \frac{T}{t}$$

AND

$$\sum_{T}^{\infty} w_t \frac{T}{t} - \sum_{T+\Delta T}^{\infty} w_t \frac{T}{t} = + w_{T+\Delta T} \cdot \frac{T}{T+\Delta T}$$

$$\therefore \quad I_T = \sum_{0}^{T} w_t + w_{T+\Delta T} \cdot \frac{T}{T+\Delta T} - w_{T+\Delta T} \cdot \frac{T}{T+\Delta T}$$

$$= \sum_{0}^{T} w_T$$

But by definition, w_t is the weight of that portion which is of correct size to settle out in time t if started from the top.

$\therefore \sum_0^T w_t$ is the summation of the weights of all those fractions which will settle out in time T or less if started from the top.

18. Proof That the Pressure at the Base of the Sedimentation Column Is a Measure of the Amount of the Material Settled Out. The only point remaining for investigation is the question of whether the change in pressure at the base of the sedimentation column is a measure of the material settled out.

Consider a column of liquid of density σ_m, cross sectional area A and height H, which has a pressure connection at the bottom.

A sample of material of weight w and density σ is added. Just before the addition the pressure was

$$p = \sigma_m \cdot H$$

The first effect of the added sample is to displace a volume of liquid equal to its own volume thus raising the liquid level and increasing the pressure. This is equal to

$$\Delta H = \frac{w}{\sigma A}$$

$$\text{Then} \quad p_1 = \sigma_m (H + \Delta H) = \sigma_m \left(H + \frac{W}{\sigma A}\right)$$

There is also another factor which increases the pressure. Since the particles are not accelerating, their weight must be supported by the liquid. Their weight is, however, only the apparent weight in the liquid or

$$w_a = w \cdot \left(\frac{\sigma - \sigma_m}{\sigma}\right)$$

This is distributed over the area A, which gives

$$\Delta p = \frac{w_a}{A} = \frac{w}{A}\left(\frac{\sigma - \sigma_m}{\sigma}\right)$$

This is the condition at the beginning of the sedimentation, before any of the sample has settled out below the pressure opening. However, it remains true during the entire process, if w_s is taken to represent the weight of the sample in suspension above the pressure opening.

$$\therefore \quad \Delta p_1 = K w_s$$

or, the change in pressure at the base of the column is directly proportional to the amount of the sample remaining in suspension. The same relation is also obtained if the problem is approached from the point of view of the change in average density due to the pressure of the sample. The original density is σ_m , the average density after the sample has been added is,

$$\sigma_{av} = \frac{\sigma_m \cdot H \cdot A + w}{A\left(H + \frac{w}{\sigma A}\right)}$$

This gives a pressure of

$$\begin{aligned} p_1 + \Delta p_1 &= \sigma_{av}(H + \Delta H) \\ &= \frac{\sigma_m \cdot H \cdot A + w}{A\left(H + \frac{w}{\sigma A}\right)} \cdot \left(H + \frac{w}{\sigma A}\right) \\ &= \sigma_m H + \frac{w}{A} \end{aligned}$$

Then $$\Delta p_1 = \sigma_m H + \frac{w}{A} - p_1 = \sigma_m H + \frac{w}{A} - \sigma_m H - \frac{\sigma_m w}{\sigma A}$$

$$= \frac{w}{A} \cdot \left(\frac{\sigma - \sigma_m}{\sigma}\right)$$

which is the same relation that was previously obtained, Therefore it can be stated:

The difference between the pressure at the base of a column during sedimentation, and the pressure at the same point due to the same height of the pure liquid without the sample, is directly proportional to the amount of the sample remaining in suspension above the point of measurement of pressure.

DESCRIPTION OF THE MICRONETER.

19. General Description. The apparatus finally constructed depends upon the principles just discussed. It consists of six sedimentation units, each equipped to measure and record the change in pressure at the bottom of the column. These units are in one housing, using a common source of time axis movement, a common liquid supply and removal system, and a common system of temperature control.

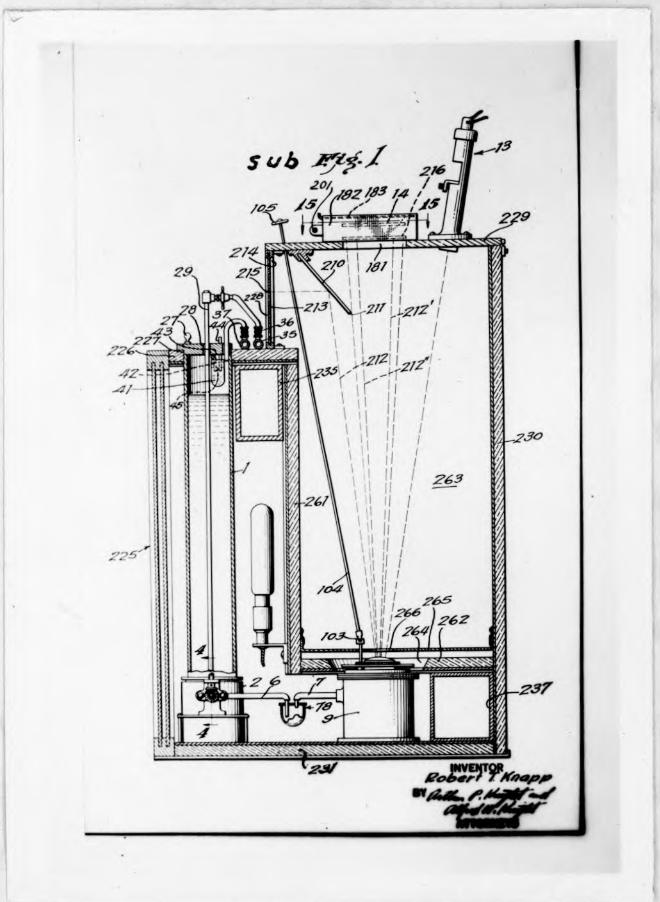


Fig. (7)

The general plan of construction of the Microneter can best be understood by referring to Fig. (7) sub Fig. (1). This is a section through the apparatus which shows the relative location of the various components of a single unit. A unit consists of the sedimentation column (1) connected to the pressure cell (9) by the tubes (6) and (7).

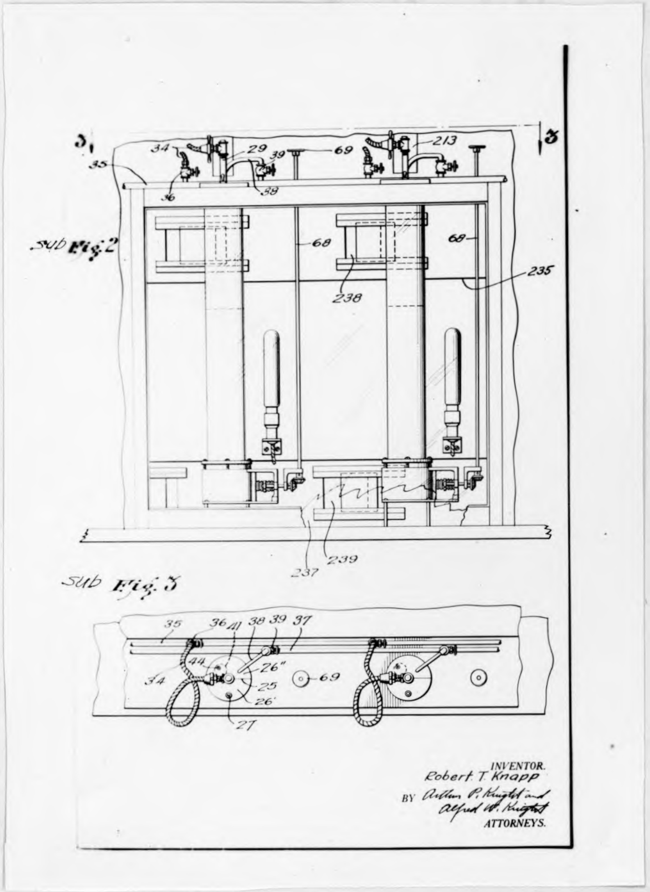


Fig. (8)

A source of light, lens and slit are contained in the small optical bench (13). A beam of light indicated by the dotted lines is sent from the bench down to the pressure cell and is reflected back up by a mirror whose position is controlled by the pressure. This reflected beam is brought to a focus on a photographic plate (14) mounted in a carriage inside the plate box (182). The shaft (201) drives the carriage and in turn is driven by an astronomical clock. Fig. (8) sub Fig. 2 shows a front elevation of two of the sedimentation columns and sub Fig. 3 shows the tops of the same.

20. <u>Sedimentation Column.</u> The sedimentation cylinder (1) is a piece of glass tubing about 3 inches in diameter by 20 inches long. It is fastened into a brass protecting ring at the top which is closed by the two halves (26') and (26") sub Fig.3 of a circular cover which fits tightly around the stirring rod (29) sub Fig.1. The front half (26') is removable to permit the insertion and stirring of the sample. This half also has in it a small hole to equalize the pressure inside the cylinder with

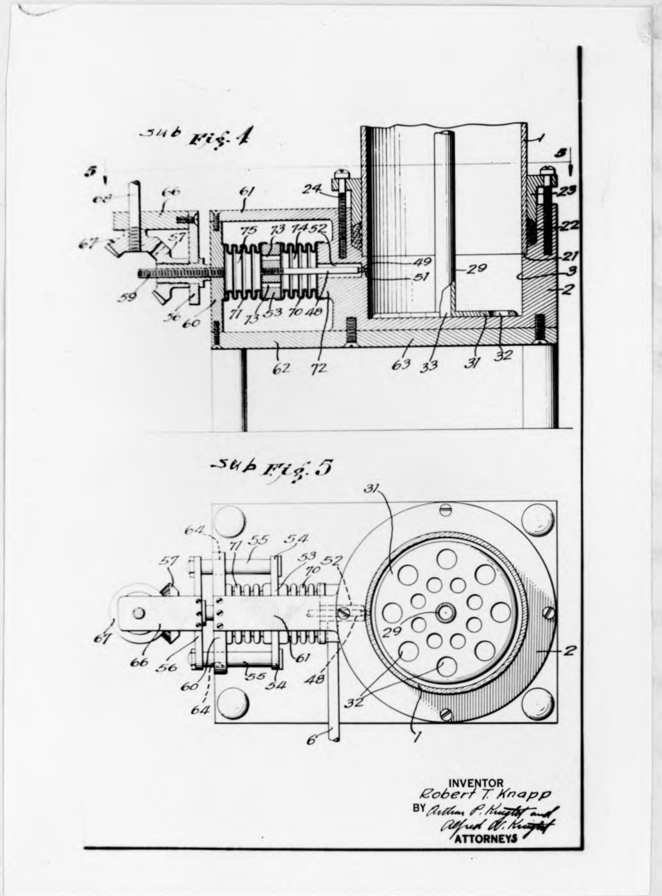


Fig. (9)

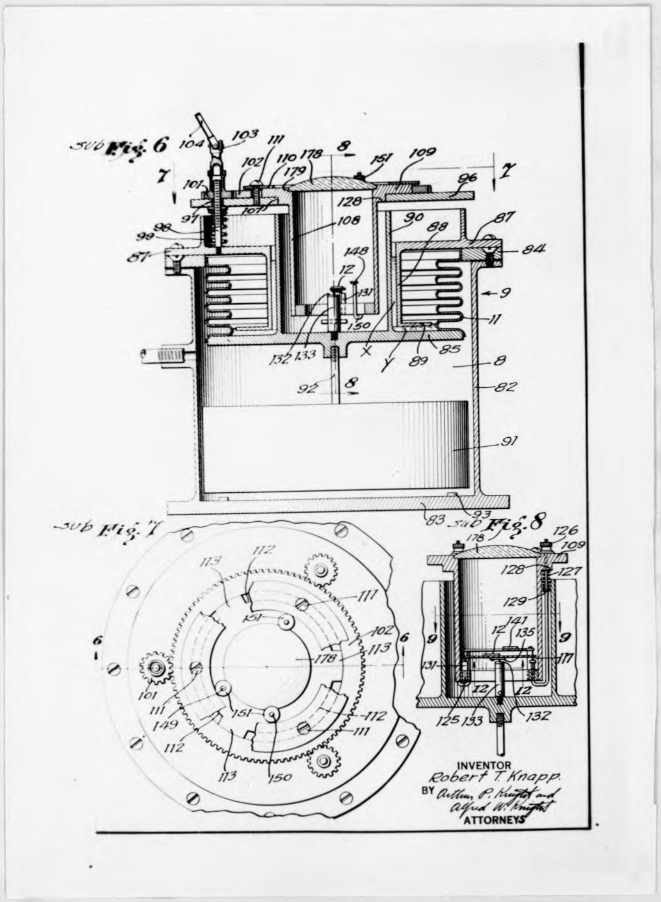


Fig. (10)

the atmosphere without permitting any appreciable evaporation of the liquid within the cylinder. The bottom of the glass cylinder is inserted into a stuffing box in the base casting. The details of this construction are shown best in Sub Fig, 4 and 5. It is seen that the stuffing box is of the conventional pattern, the packing used (22) is a series of rings cut from sheet cork and dipped in shellac as they are put in place. This makes a joint that gives no trouble. The pressure opening is at the point (51) sub Fig 4 and is quite close to the bottom of the cylinder. This opening is closed while the sample is being introduced and mixed by means of the special valve shown in sub Fig. 4 and 5.

21. Sedimentation Column Valve. The purpose of this valve is to provide a means of isolating the sensitive pressure element from the

rather violent disturbances incident to the introduction and removal of the sample, and at the same time to avoid joints that might allow any liquid to escape. This is done by sealing the valve stem inside two sections of a small metal bellows (71) and (72) and operating it from the outside by the mechanism shown. The purpose of providing two sections of bellows is to eliminate any net change in the volume of the system by causing one section to expand when the other section is compressed during the operation of the valve. This is desirable in order that the pressure element will not displace the light beam from its correct position. The valve is controlled by the handle (19) shown in sub Fig.3. The tube (6) sub Fig.5 connects the valve with the pressure element.

22. Stirring Rod. The construction of the stirring rod can also be seen in sub Fig.4 and 5. It consists of the tube (29) and the perforated plate (31). Stirring is accomplished by moving the rod up and down. It was found that this device secured the most thorough mixing with the least persistent eddy currents.

23. Settling Chamber. It is desirable to prevent any possibility of particles of cement getting over into the pressure cell through the connecting tube. Therefore a small settling chamber (78) sub Fig.1 is provided between the sedimentation column and the pressure cell. This is purely a settling chamber and not a filter due to the necessity of not introducing any resistance to flow.

24. Pressure Unit.(16) The construction of the pressure unit

is shown in sub Figs 6 to 12. It is composed of a cylindrical chamber (82) sub Fig.6, whose top is closed by the metal bellows (11).* Since this bellows is very flexible, a change of pressure in the chamber will cause it to move. It will compress with an increase in pressure and expand with a decrease. Since it should be sensitive to very small changes in pressure it is undesirable that the total pressure due to the column of liquid in the sedimentation cylinder should be resisted by a deflection of the bellows. If this were the case a bellows with a high spring constant would be required, which would decrease the sensitivity. Therefore the weight of the column of liquid is counterbalanced by means of the weight (91) sub Fig.6. This is so adjusted that the weight is sufficient to counterbalance about 98 $^o/o$ of the total liquid column.

A small pedestal (132) is fastened to the bottom of the bellows. The top is covered with the brass plate (133). A ring of brass (87) is fastened to the top of the unit, and this in turn carries a second ring (96) supported by the screws (98) and springs (99). The height of this ring (96) above ring (87) is adjusted by means of the ring gear (102) sub Fig.7 and pinions (101) through the universal joint (103) and (104) and handle (105) shown in sub Figs. 1 and 7. This ring (96) carries an assembly which contains a lens (178), a fixed mirror (141) and two bearing points (122) for a movable mirror (12). Various views of the details may be seen in sub Figs. 4 to 11. A bottom view of the

(* The present units use a bellows long enough to entirely fill the chamber. The weight (91) is on top of plate (85) instead of suspended from it. However, the action is the same and the rest of the construction is unchanged.)

movable mirror mounting is shown in sub Fig.12. This mounting rests on the three needle points (116),(117) and (118). Points (116) and (117) rest in cups composed of a watch ring jewel on top of a cap jewel (122) which is mounted on the assembly carried on ring (96). The adjustable point (118) rests on the glass plate (133) which moves with the bellows. Therefore it is seen that when the bellows moves vertically the mirror (12) is rocked about a horizontal axis joining the points (116) and (117). The initial angle of the mirror is determined by the position of the ring (96) which is controlled by the handle (105).

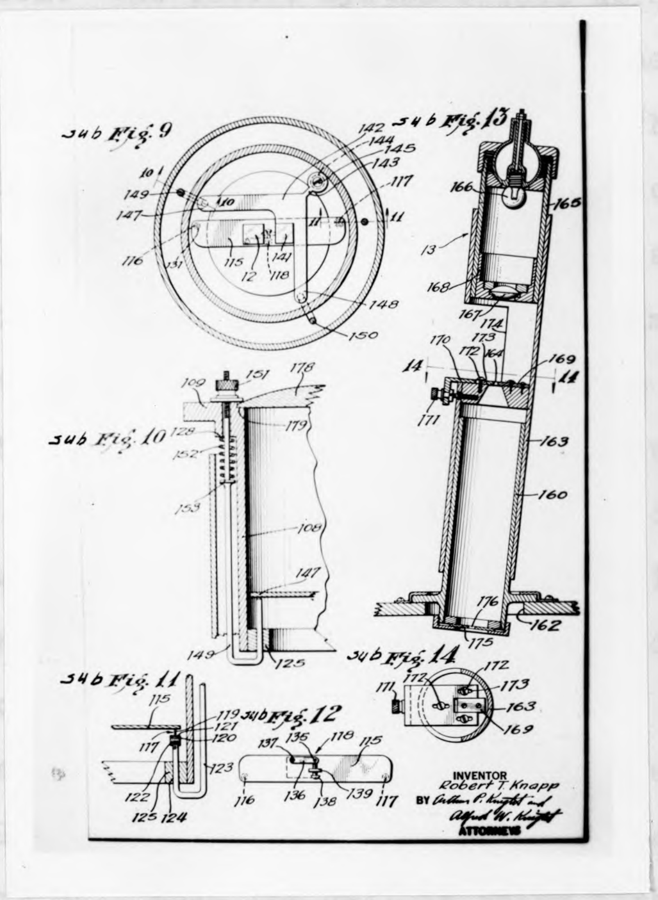


Fig.11

25. Optical System. The component parts of the optical system are best seen in sub Fig.1. They are the optical bench (13), the mirror and the lens described above, the cylindrical lens (216), the mirror (210), and the ground glass (213). The details of the optical bench are shown in sub Fig.13. The light (166) is a concentrated filament flash light bulb, receiving current from a storage battery. It is mounted in a ball and socket receptacle to facilitate setting it in correct position so that the image of the filament will fall across the slit. The lens

(167) has a short focus so that the image of the filament may be sharply focused on the adjustable slit (164)(See also sub Fig.14) thus causing the slit to become the source of illumination. The entire system slides up and down on the table (160) thus making it possible to bring the image of the slit into sharp focus on the photographic plate. The aperture (176) is a rectangular opening,adjustable laterally to allow the light beam to illuminate the mirrors in the pressure cell, but at the same time to eliminate stray light that might be reflected from the other parts of the cell and fog the plate. Sub Figs 6,8, and 9 show the optical parts of the pressure cell. The lens (178) is a simple plane-convex lens of 1.37 dioptors focus. The mirror (12) is the recording mirror whose mounting has already been described (par.24). The mirror (141) is fixed and is used to produce a horizontal zero line on the plate. The beam from this mirror may be adjusted in both the x and y directions by the rods (149) and (150) which tilt the mirror mounting. Such adjustment is necessary only when the cell is being first placed in operation to bring the light spot to the correct location on the plate. For the same reason, one of the "fixed" jewel cups for the recording mirror is adjustable as shown in sub Fig.11. These mirrors are of "Stellite", ground and polished optically flat, and have been found very satisfactory for the work. The cylindrical lens (216) , sub Fig.17 and 18, has a focus of about 3 cms. and serves to sharpen the image of the slit on the photographic plate. The mirror (210) sub Fig.1 serves to intercept the beam from the mirror (12) when it is too far to the left to fall on the plate, and cause it to appear instead on the ground glass screen (213).

This is used to bring the liquid to the correct level in the sedimentation column (1) before the sample is added.

26. Movement of the Pressure Cell. It is interesting to calculate the motion of the pressure cell necessary to cause the full deflection of the point of light on the plate. Full scale motion is 6.54 cm. The distance from the mirror to the plate is about 75 cm. The separation of the fixed and movable needles on the mirror averages 0.25 cm. Therefore the movement of the needle support necessary to cause full scale deflection is :

$$\Delta l = \frac{0.25}{2} \times \frac{6.54}{75} = .0087\,\text{cm.}$$

27. Plate Box. The details of the plate box may be seen in sub Figs. 15,16, and 17. It is an aluminum casting with a light tight slide that covers the entire top. The plate holder or carriage (185) is a simple aluminum frame with a recess (193) to hold the standard lantern slide used. The plate is held in place by spring clips. The carriage is guided by four rollers (186) which bear on the drill rod rails (184). The carriage is driven by the half nut (194) which engages with the screw (196). The screw threads are

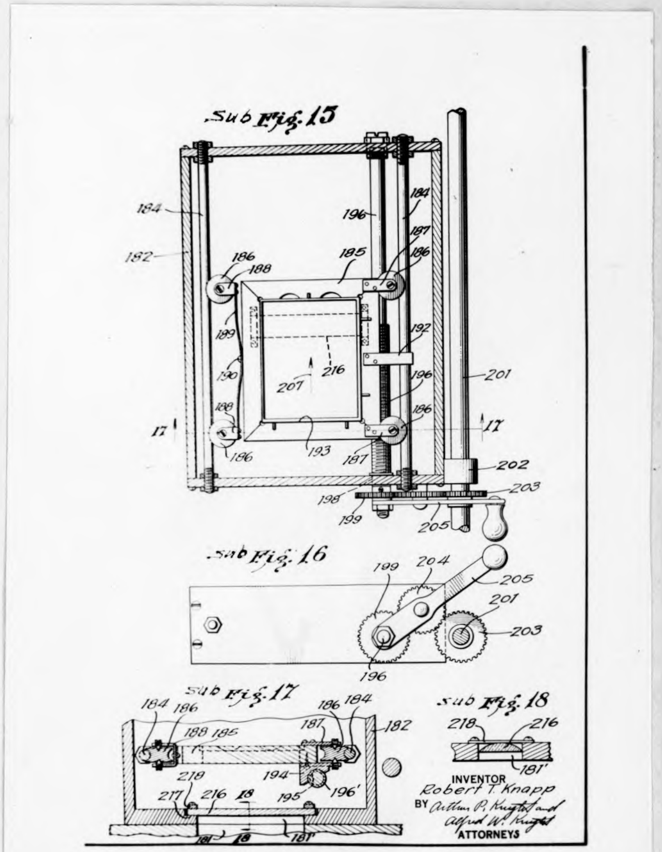


Fig. (12)

of such length that when the carriage comes to the end of its travel, the nut runs off the end of the thread and the carriage stops. It is returned to the starting position by compressing the spring (189), lifting that side of the carriage and sliding along the other rail. The screw thread (196) is driven by the shaft (201) throught the gear train (199)(204)(203). The gear shift lever (205) allows gears (204) and (201) to be placed in mesh or to be left disengaged.

28. Drive Clock. The shaft (201) is directly connected to the five minute shaft of an astronomical clock. The pitch of the screw (196) is 10 threads per inch. The gears (196) and (203) are the same diameter. Therefore the speed of the plate is one inch in fifty minutes. Unit No.6, however, is provided with several sets of interchangeable gears so arranged that several speeds can be obtained, thus making it possible to vary the time required for the total travel of the plate from one half hour to twenty-four hours. The clock housing and stack of driving weights are seen in Fig. (13).

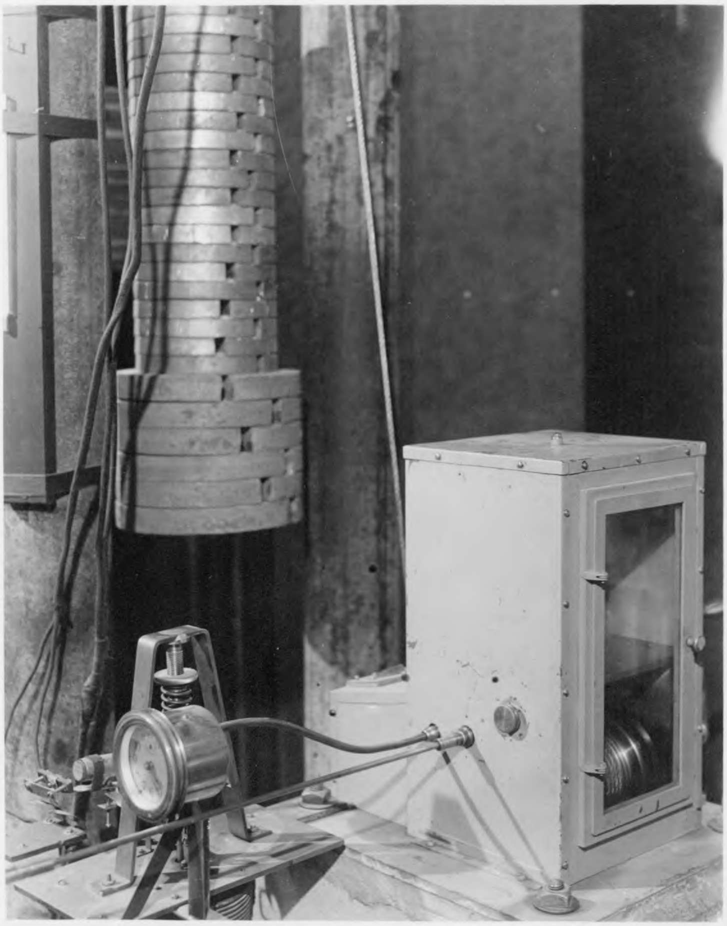


Fig.(13)

Although this is a very large amount of weight for a clock of this size and although the load is heavy and variable, depending on the number of units in operation, the clock has been very satisfactory. Its rate is very constant. Deviations are so small that they cannot be detected with the means available; namely, high grade pocket watches checked daily against Western Union Time. Since all of the plate carriages of the different units are driven from the same shaft the time axes on all the plates are identical.

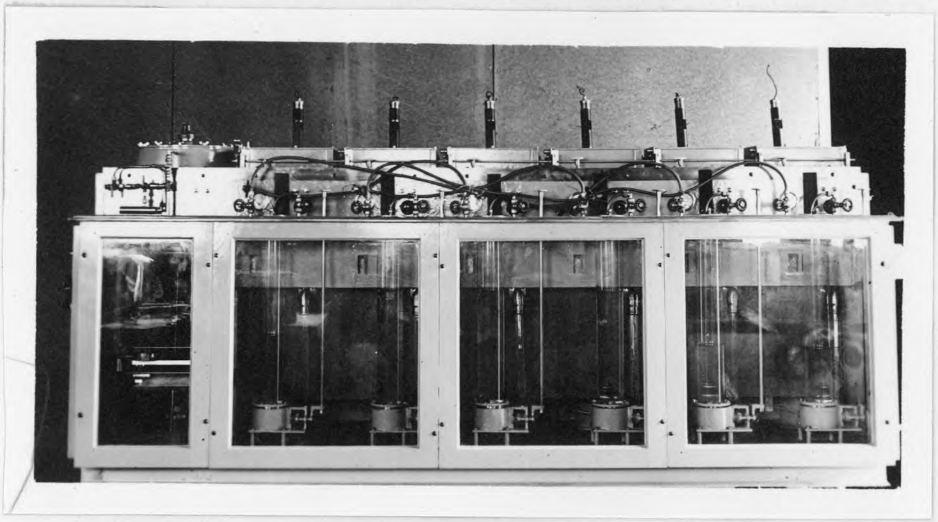


Fig. (14)

29. <u>Housing of Apparatus</u>. The six units are housed in one case, as shown in Fig.(14). The frame is made of channel iron welded together to give a very rigid construction. The front, where the sedimentation columns are located, is closed with double glazed windows to decrease the heat leakage. The top is of transite board and the sides and bottom are covered with "Masonite", a fibrous insulation board. The partition (261) sub Fig.1 prevents any light coming through the front windows from reaching the mirrors or plates. The apparatus is grou~~ted~~nded onto a four inch cushion of cork, which in turn is grouted onto the floor. This serves to reduce the effects of external vibrations.

30. Temperature Control. The entire apparatus is kept at a uniform temperature by a rapid circulation of air. The ducts are shown in sub Fig.1. The inlet is (235) and the outlet is (237). The air is distributed uniformly by small openings in the ducts between every unit. The inlet duct and openings may be seen in Fig.(14). The air coming out passes through a heating chamber containing Ward-Leonard heaters manually controlled, and a base resistance heater controlled thermostatically. From the heating chamber the air passes into the suction of a blower which delivers it to the inlet duct. The system is completely closed, using the

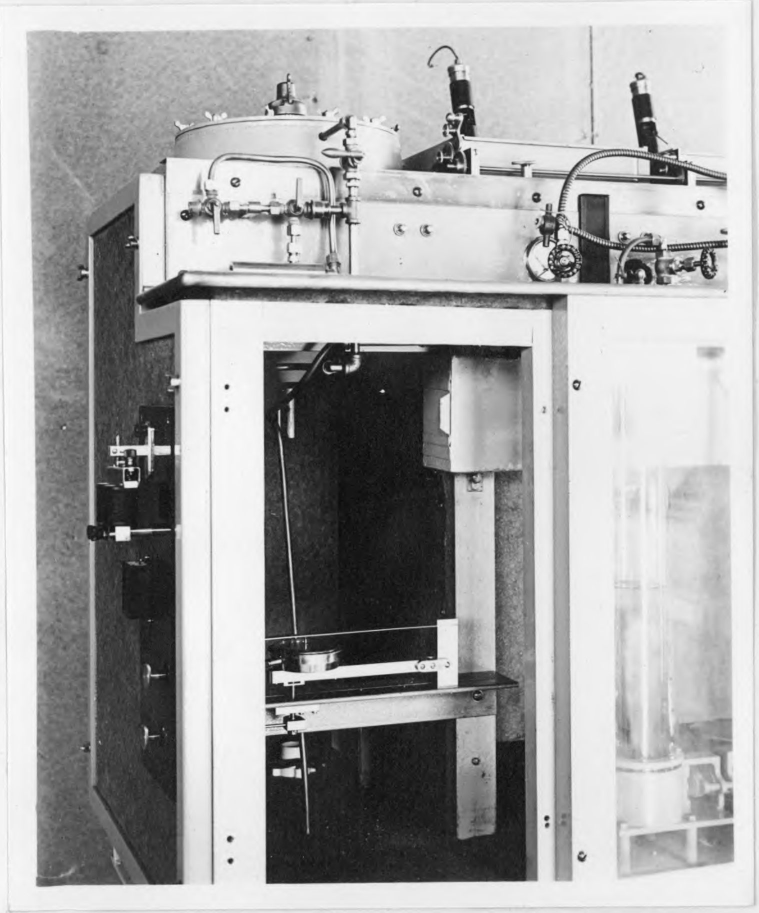


Fig. (15)

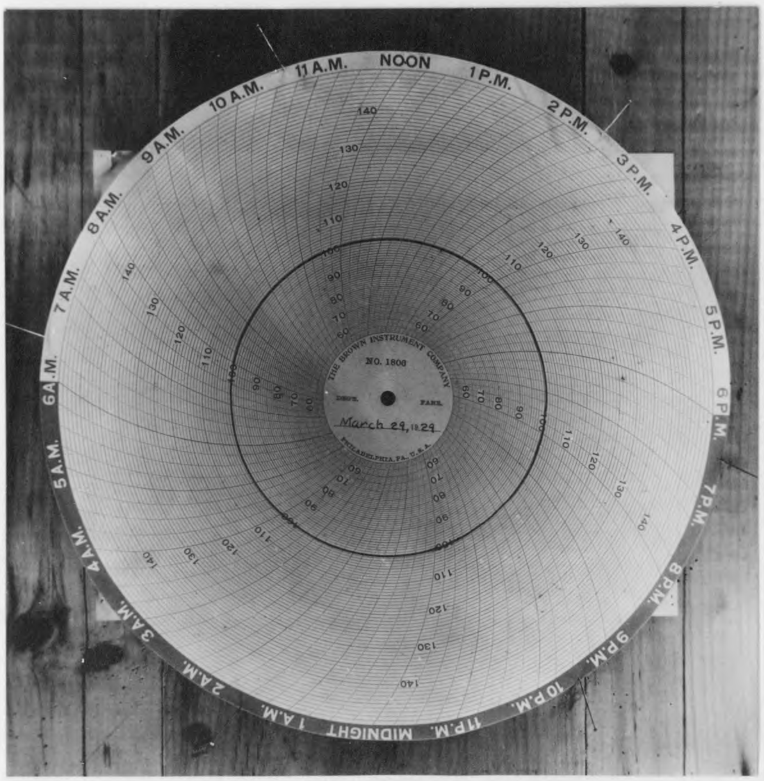


Fig.(16)

same air continuously. This avoids trouble with dust, which whould certainly be encountered if fresh air were used, and also saves heat. The bare coils in the heating chamber are controlled by a bimetallic strip mounted inside the apparatus. This may be seen in Fig.(15) together with the relay which is mounted on the outside of the case. The contact with the bimetallic strip is made by a platinum point mounted on the hour shaft of a small 8-day clock. This slow turning effectually breaks any minute welds at the contact points and makes the regulation very close. The two knobs seen in the lower left hand corner of Fig.(15) allow the clock to be wound and the temperature regulated without opening the apparatus. Fig.(16) shows a photograph of the daily temperature record taken near the outlet duct.

Fig.(17)

31. Liquid System.

In the operation of the Microneter it is necessary to have a supply of liquid of the correct temperature and also a removal system to take care of the liquid in the sedimentation chambers after analyses are completed. To meet this first requirement a five gallon storage bottle is placed inside the apparatus, thus insuring a supply of liquid at the corect temperature. The bottle is arranged so that air pressure can be applied to it and a system of piping carries the liquid to each sedimentation chamber. Thus by opening a valve the desired unit can be filled with fresh liquid. The used liquid may be removed by either one of two methods. A suction tube may be inserted into the sedimentation chamber and the liquid lowered to any desired level. Or, since the stirring rod is hollow suction may be applied to it to remove the liquid. Fig. (17) shows the supply lines to each unit coming into the top from the right. It also shows the flexible connection used to apply suction to the stirring rod. It is seen that each unit has control valves for both supply and suction lines. Fig. (15) shows the air aspirator used to provide the suction required . It is seen in the upper left hand quadrant.

32. Dark Room. In order to facilitate the operation of the Microneter it was placed inside a dark room. This was constructed with a double wall of "Masonite". The space between was filled with shavings. The entrance is through a labyrinth. The room is kept at a uniform temperature by means of a thermostat, operating with a range of two degrees Fahrenheit.

33. Viscosimeter. A viscosimeter of the capillary tube pipette type is mounted inside the Microneter. It is arranged so that it can be operated from the outside to avoid disturbance of the temperature equilibrium. The pipette may be seen behind the window in Fig. (18).

34. Density Measurement. A Chain-o-matic balance is mounted on one end of the Micrometer, as seen in Fig. (18). This is used to do all of the necessary weighing of samples, residues,etc. It also is used to determine the densities of the liquid and of the samples. A small hole is drilled under one pan and down into the Micrometer itself. A piano wire which passes through the hole may be fastened to the balance pan. Weighings may then be made inside the machine by attaching the object whose weight is desired to the wire. Thus the liquid densities are determined by attaching an Invar density-plummet to the wire and weighing it immersed in a beaker of the liquid. Sample densities are determined by hanging a small bucket from the wire. A small amount of the material is placed on the bucket and weighed. The bucket is them immersed in a liquid whose density has been determined, and weighed again. Obviously, the sample density can be calculated from these two weights.

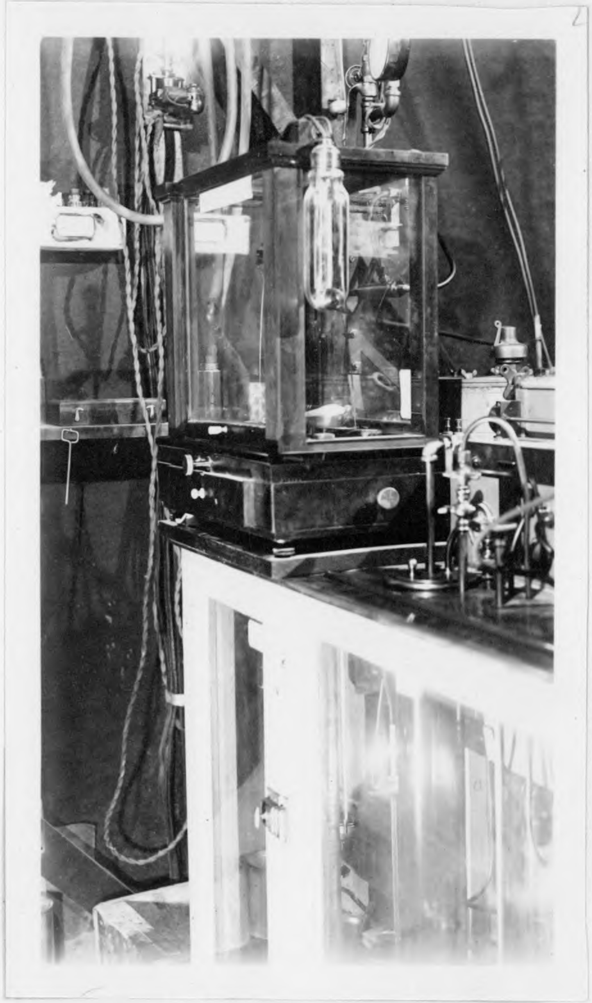


Fig. (18)

35. Choice of Liquid. There are several factors that govern the choice of the liquid to be used. The density and the

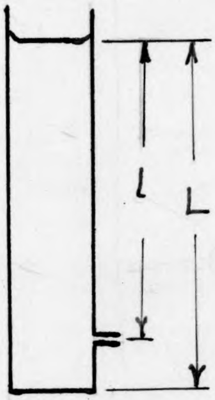
viscosity are factors affecting the rate of fall of the particles. These properties also determine the maximum size of particles that may be handled without deviation from Stokes' Law. This will be discussed (Par. 43 e). Another factor to be considered is the effect of the liquid on the material to be analyzed. There are two different ways in which the liquid and the sample may interact. First, the material of the sample may react chemically,with the liquid,thus changing the size and properties of the individual particles. Thus water obviously is not a satisfactory medium to use with a sample of cement. Second, there may be a physical reaction between the two. This is the case when flocculation occurs. The term flocculation is used to designate a condition in which a finely divided substance distributed throughout a liquid is found to form in groups of a large number of particles apparently held together by some attraction forces. Of course there is no possibility of determining the size distribution of the individual particles under such conditions. It is necessary,then,in choosing a liquid to avoid selecting one that either reacts chemically with or possesses the property of causing flocculation of the materials to be tested. However, the causes and the preventatives of flocculation are rather obscure. Therefore the best course seemed to be to test various liquids that were acceptable from thechemical point of view to see whether or not they caused flocculation. The workers abroad had generally adopted absolute alcohol as the standard liquid for the use with cement, with the addition of a small amount of calcium chloride as a deflocculator. However, it was felt that alcohol would not be desirable for our use because of the difficulty of obtaining it in quantity and the greater difficulty of maintaining it in a water-free condition. Petroleum distillates seemed to offer a series of substances possessing desirable viscosities and densities. They also showed no chemical activity with cement. A number of them were tested for flocculation. The lighter ones, benzine,gasoline and high grade kerosene caused heavy flocculation. Low grade kerosene was found to be quite variable. One sample would show no flocculation while another of the same brand purchased at a different time produced bad conditions. Finally a sample of stove distillate was tested. This is a very low grade distillate which has a dark brown color. It showed absolutely no signs of flocculation irrespective of the concentration of cement. The only drawbackto its use was its viscosity, which was so high that it would necessitate a longer time of run than was convenient if the analysis was to furnish information down to a predetermined minimun size. However, it was found that when mixed with kerosene it imparted its non-flucculating properties to the mixture and, at the same time, an acceptable viscosity could be obtained. Therefore this was adopted as the standard liquid for use in the Microneter.

TECHNIQUE OF OPERATION.

36. Calibration of the Pressure Unit. It is necessary to have a calibration to each pressure unit to be able to calculate the necessary sample weight. This is accomplished by adding definite volumes to the sedimentation liquid and observing the

corresponding deflection of the spot on the plate. For this purpose a set of standard rods was constructed. The volume of each rod was carefully determined. They are suspended in turn in the liquid by means of fine wires. The immersion of one of the rods in the liquid is exactly equivalent to the addition of a corresponding volume ofliquid as far as producing a pressure change at the bottom of the chamber is concerned, and is much more accurate. Figs. (19),(20),(21) are a set of typical calibration curves taken at random. They show how nearly a perfect linear relation exists between the volume displacement(and therefore the pressure change) and the deflection on the plate. These curves are perfectly reproducible. It should be noted that this calibration is an overall one, i.e., it includes all errors that may occur in the optical system, pressure unit, and connections to the sedimentation chamber itself.

37. <u>Calculation ofCorrect Weight of Sample.</u> The sample weight is then calculated in the following manner:



Let the constant ofthe unit be defined as

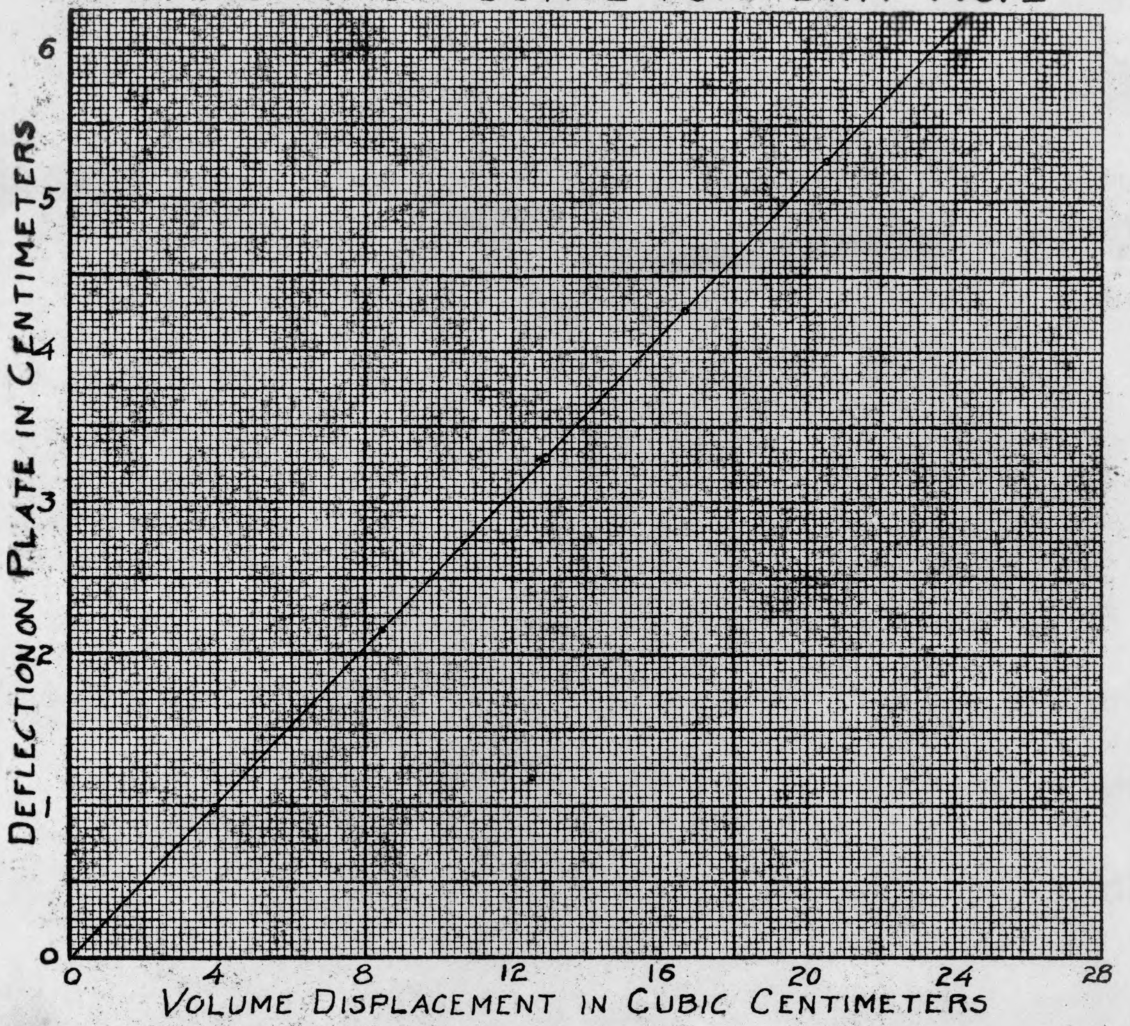
$$K = \frac{\text{Deflection on plate}}{\text{Volume displaced} \times \sigma_m}$$

where W_s = wt. of sample
σ = density of sample
σ_m = density of liquid

$$\text{Then} \quad W_s = \frac{\text{Deflection desired}}{K} \cdot \frac{L}{l} \cdot \frac{\sigma}{\sigma - \sigma_m}$$

The reason for the term $\frac{L}{l}$ is that the portion of the sedimentation chamber below the pressure opening is dead space as far as the analysis is concerned, serving only as a space where the sample collects after it passes the pressure opening. However since the sample is

CALIBRATION CURVE FOR UNIT NO. 1



CALIBRATION CURVE FOR UNIT NO. 2

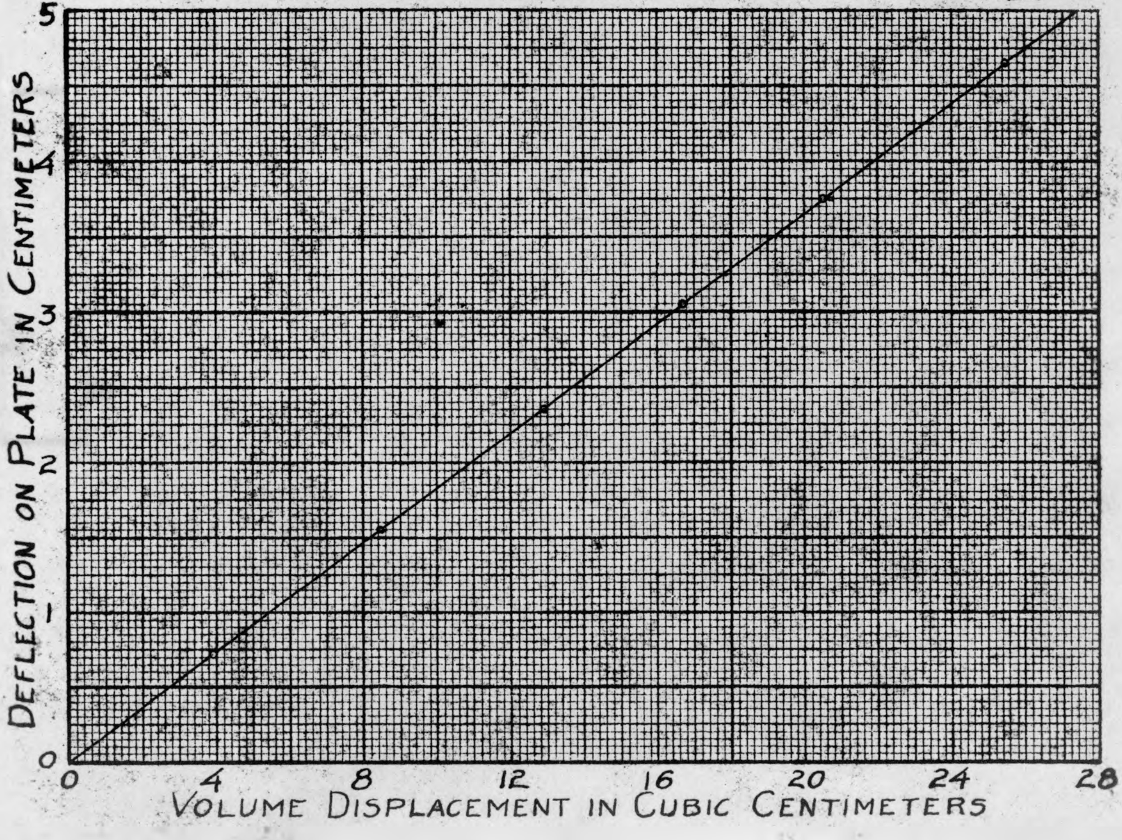
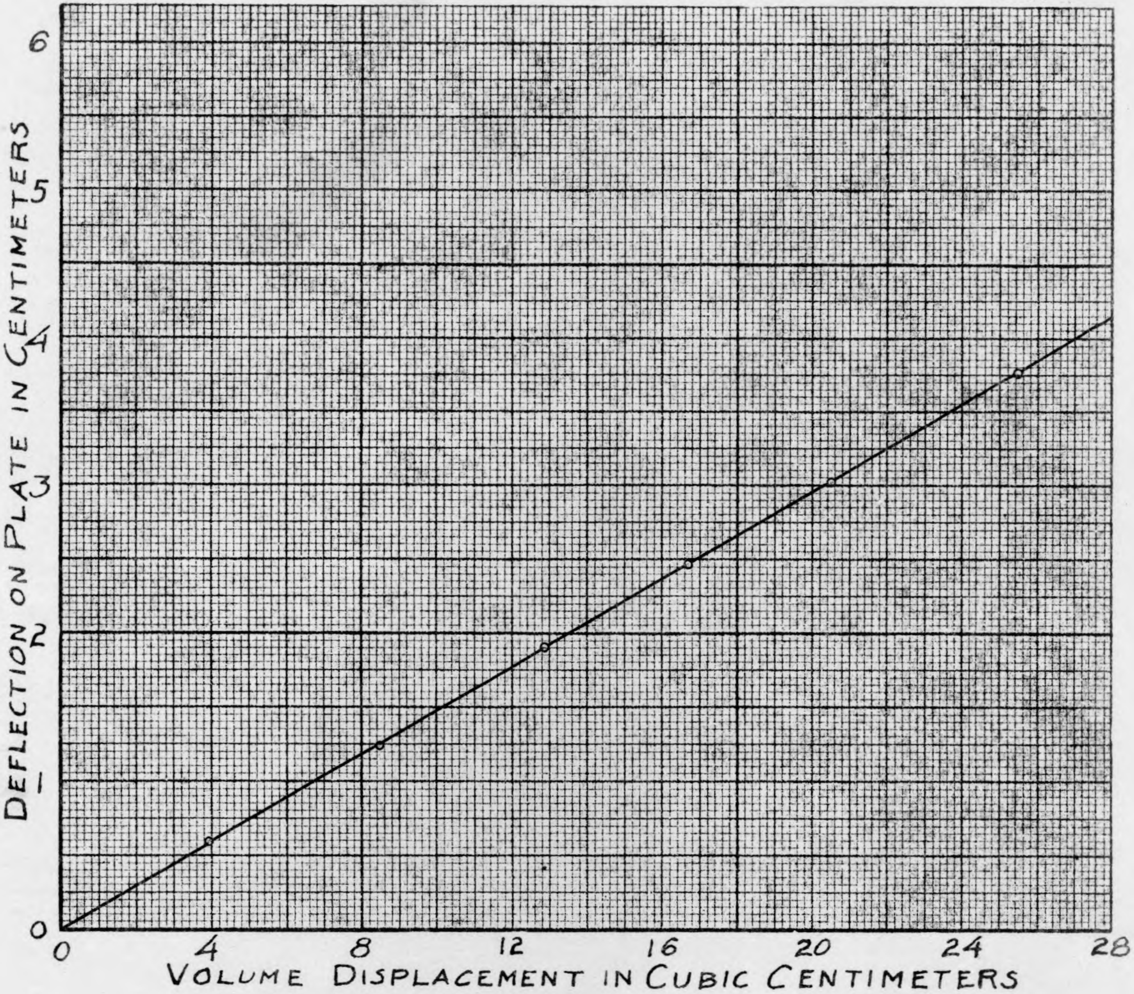


FIG. 19

CALIBRATION CURVE FOR UNIT NO. 3



CALIBRATION CURVE FOR UNIT NO. 4

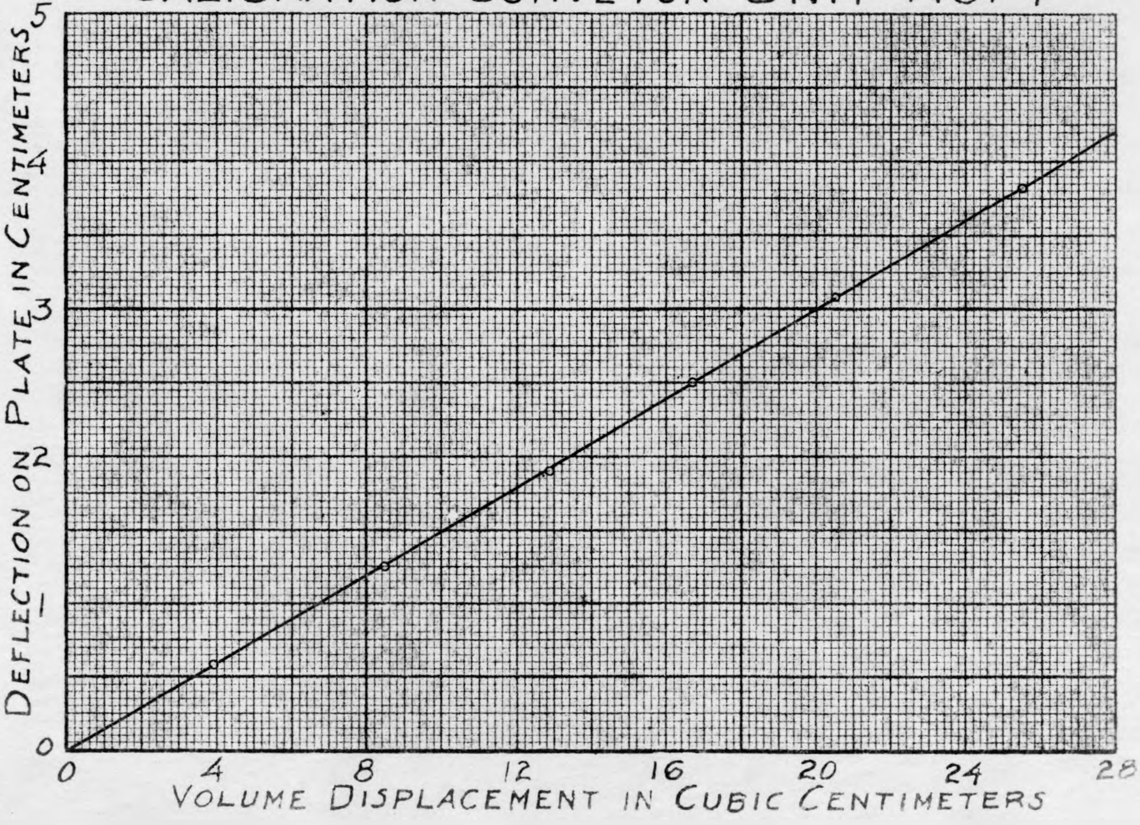
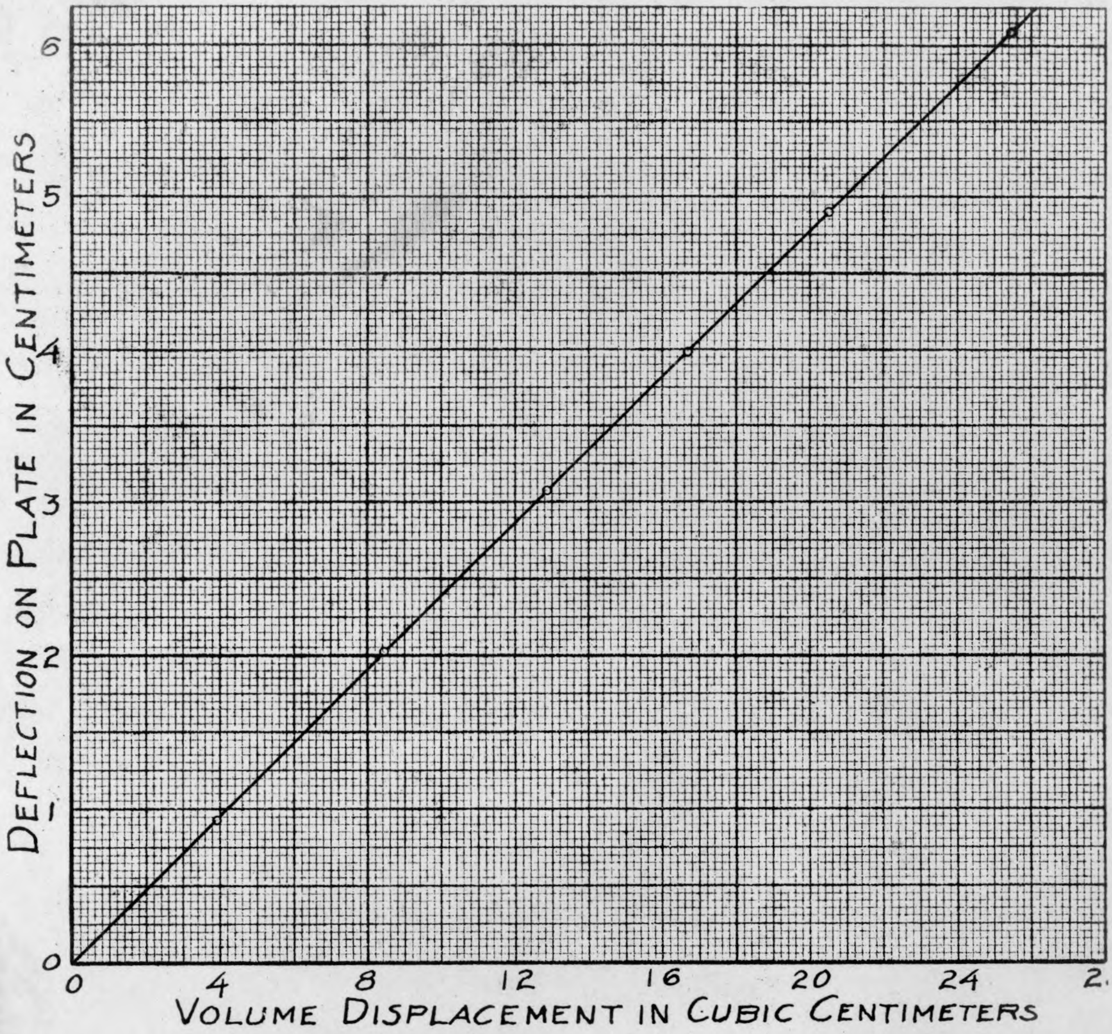


FIG. 20

CALIBRATION CURVE FOR UNIT NO. 5



CALIBRATION CURVE FOR UNIT NO. 6

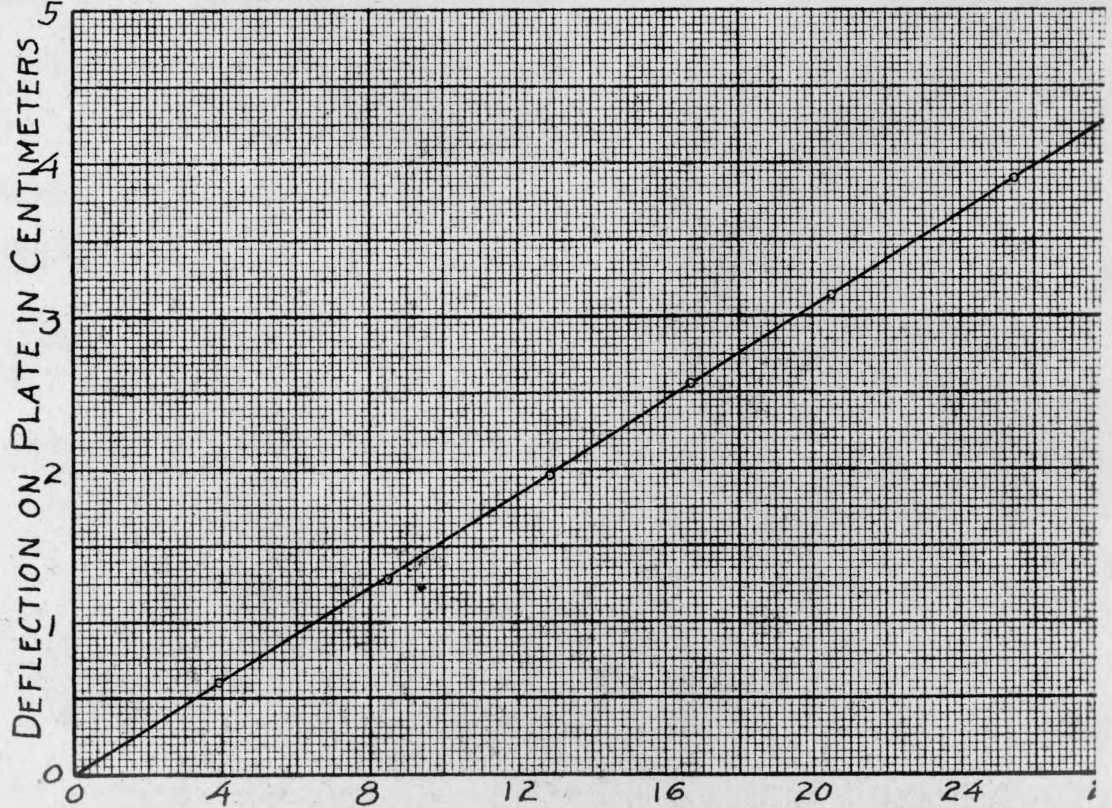


FIG. 21

is uniformly distributed at the beginning there is a proportionate part ofit in this space. Therefore the original sample must be of such a size that, at the beginning of the analysis,there will be the correct amount present above the pressure opening to produce the full desired deflection when it all settles out. Each unit has its individual sample weight which is so calculated that they all produce exactly the same deflection on the plates. This deflection has been chosen to be 65.4 mm because when placed in the projector it gives a deflection of 200 mm on the screen.

38. Determination of the Initial Liquid Level. Since the sample has volume it causes an additional deflection other than that due to the increase in density of the mixture. The amount of the deflection is constant, no matter whether the sample is uniformly distributed throughout the liquid or settled on the bottom; therefore it is not desirable that it should appear on the plate. To prevent this the liquid level is adjusted so that the spot of light will just come to rest at 65.4 mm deflection from the zero point when a rod whose volume is equal to that of the sample is suspended in the liquid. The rod is then withdrawn,allowed to drain back into the liquid, and the position of the spot,which now appears on the front ground glass window, is marked. This is used as an index when the chamber is filled with fresh liquid. Liquid is added until the spot is at the index mark. This is a very accurate method of setting the liquid level, for the spot is sensitive to about three drops of liquid.

39. Selection of the Sample. It is axiomatic that the analysis can be no better than the sample. If the sample is not truly representative of the material the analysis is equally inaccurate; therefore the method of sampling was carefully considered. The procedure adopted is as follows: (a) When the material is obtained during the operation of a large sized mill, the sample brought to the laboratory is a composite of many small samples taken at short uniform intervals from a moving stream of material. Care is taken that the samples are obtained from a complete cross section of the stream so that any tendency to size classification due to the motion will introduce no error. (b) When the material to be analy~~si~~zed is the product of laboratory jar mill run, a sample representing a large portion of the total charge is saved. It is obtained by riffling the entire charge through a standard sample splitter or riffle. (c) In either case the further treatment of the sample is the same. It is again reduced in size by means of the riffle until an amount is obtained that will yield, when sieved, enough -200 mesh material for a Microneter analysis. This material is then sieved through standard nest of sieves on a "Rotap" for 15 minutes. Care is taken not to overload the sieves. If one sieving does not yield enough -200 mesh material the whole process is repeated until enough is obtained. The results of each sieving are recorded. The figure used as the percentage of -200 mesh material is the weighted average of all the sievings made to secure the Microneter sample. When the material being sieved contains an appreciable amount of very fine

material that shows a tendency to stick to the larger particles or to the sieves, these sieves are shaken by hand to completion, after the Rotap. The -200 mesh material obtained is stored in small numbered tin cans with tight fitting covers. (d) When the sample is to be run in the Microneter this can is rolled to mix the material, then opened and stirred thoroughly. The correct amount of material for the Microneter unit to be used is weighed out on the Chain-o-matic balance. The average sample weight is about 35 grams and the maximum error in weighing is plus or minus 2 milligrams. The sample is stored in glass vials at the unit until time to start the run.

40. Microneter Procedure During Running of Sample. A brief description of the running of a sample is given below. (a) The sedimentation chamber is cleaned from the last run,refilled with liquid to the correct level and allowed to stand for at least 20 minutes to reestablish equilibrium. (b) The liquid level height is then readjusted by means of a medicine dropper until the spot of light is at the cofrect position on the front ground glass. The light is then turned off. (c) The room is darkened and the plate carriage is loaded with a correctly labeled plate. The plate box cover is replaced and the rest of the operation is carried on with normal illumination in the room. (d) The valve between the sedimentation chamber and the pressure unit is closed and the sample is emptied into the chamber. (e) The sample is stirred vigorously for at least two and one half minutes. (f) The plate carriage gear train is then thrown into mesh and the light in the optical bench turned on. (g) A few more strokes are made with the stirring rod and the valve

is opened immediately after the completion of the last stroke. The stirring rod remains in position throughout the run. (h) Nothing is disturbed for three hours and fifteen minutes. At the end of this time the recording light is turned off, the valve is closed and immediately the suction tube is placed in the chamber and the liquid is removed down to the level of the pressurem opening. (i) The plate is removed and stored while awaiting development, and the unit is ready for the next sample.

41. Method of Obtaining Zero Settings for Plate. The liquid that was withdrawn is next filtered to determine the amount of material left in suspension above the pressure opening. Quantitative filter papers are used for this purpose and the liquid is filtered through twice. The paper is then drained and burned. The oil remaining in it supplies sufficient fuel to insure that the paper is completely consumed. The residue is then weighed. The ratio of this weight to the weight of the original sample that was above the pressure opening is the fraction of the active material that has not settled out. This gives a definite location in the "y" direction for the end of the curve. The fraction, expressed as a percent, gives the distance below the one hundred percent line that the curve should be at the end of the three hours and fifteen minutes. This is used to set the image of the plate in the projector when the plate is being analyzed. This procedure will be discussed later (Par.48)

42. Temperature,Viscosity, andDensity Control. The effects of changes of temperature,viscosity, and density of the liquid upon the operation of the Microneter are interrelated. The tempera-

ture effects both the viscosity and the density. However, it is seen from Stokes' Law

$$v = \frac{2}{9} g \frac{(\sigma - \sigma_m)}{\eta} r^2$$

that both may vary without affecting the rate of fall of the particles, provided that the ratio $\frac{\sigma - \sigma_m}{\eta}$ is kept constant. Advantage of this fact is taken in the operation of the apparatus. The general procedure is to hold the temperature, viscosity, and density at standard values. However, when a new batch of liquid is made up, or when the season of the year makes it desirable to establish a new level of temperature it is necessary to readjust the values. By varying the ratio of stove distillate the kerosene and the temperature it is possible to keep the ratio $\frac{\sigma - \sigma_m}{\eta}$ constant. It should be noted that such changes are made at relatively long intervals and that the conditions are kept constant over long periods of time; for example, after 500 analyses. A typical set of conditions that were maintained for some time was, temperature 100° Fahr., density 0.820 g. per cc, viscosity 0.01814 poises

CHECKS UPON THE OPERATION OF THE MICRONETER.

43. Theoretical Limitations. During the development of the Microneter, it was desirable to employ several methods of checking the accuracy of performance. One of these was to determine whether or not the conditions of operation were such that the settling particles could be expected to obey Stokes' Lae. Reference was made to the discussion of this law found in Dr. R.A. Millikan's book "The Electron".(17) In it he gives the following five assumptions as the ones used in the theoretical derivation: (1) that the inhomogeneities in the medium are small in comparison with the size of the sphere; (2) that the sphere falls as it would in a medium of unlimited extent; (3) that the sphere is smooth and rigid; (4) that there is no slipping of the medium over the surface of the sphere; (5) that the velocity with which the sphere is moving is so small that the resistance to the motion is all due to the viscosity of the medium and not ~~all~~ due to the inertia of such portions of the medium asare being pushed forward by the motion of the sphere through it.

(a) First Assumption. The first assumption is certainly satisfied in the case of the Microneter, since the distances between the molecules of a liquid are of the same order as their molecular diameters, and therefore very small in comparison to a particle whose diamtere is a micron or more.

(b) Second Assumption. The degree to which the second assumption is fulfilled depends upon the dimensions of the sedimentation cylinder and their relations to the size of the particle. This has been investigated by Ladenburg, who reports that the walls introduce an effect which changes the simple Stokes' Law to read:

Where R = radius of cylinder
L = length

$$V = \frac{2}{9} \cdot \frac{g r^2 (\sigma - \sigma m)}{\eta (1 + 2.4 \frac{r}{R})(1 + 3.1 \frac{r}{L})}.$$

This causes our equation III page (14) to become

$$r = \frac{K' \sqrt{(1 + 2.4 \frac{r}{R})(1 + 3.1 \frac{r}{L})}}{\sqrt{T}} \text{ instead of } r = \frac{K'}{\sqrt{T}}$$

It is seen that the value of this correction term is greatest where r is a maximum. Since our largest particles have a diameter of 90 microns (i.e., $r = .0045$ cm) if this value is substituted for r the maximum correction will be obtained. Thus

$$\begin{aligned} \sqrt{(1 + 2.4 \tfrac{r}{R})\ (1 + 3.1 \tfrac{r}{L})} &= \sqrt{(1 + 2.4 \tfrac{.0045}{3.5})(1 + 3.1 \tfrac{.0045}{46.6})} \\ &= \sqrt{1.0031 \times 1.0003} \\ &= 1.0017 \end{aligned}$$

Hence our deviation from the second assumption can introduce, at most, an error of 0.17 per cent.

(c) Third Assumption. The third assumption is, of course, not satisfied. The particles are not smooth, rigid spheres. However, they do average about the same shape. It has been found that the effect of this is to cause the diameter obtained by calculation from Stokes' Law to be different from the mean diameter of the particle. This error is eliminated by the introduction of a "shape factor" which has to be obtained by calculation. The determination of this factor will be discussed later at some detail (par.44).

(d) Fourth Assumption. The fourth assumption offers no difficulties. Millikan quotes the work of Dr. H.D. Arnold, who used spheres of rose metal of diameters from 40 to 2000 microns, settling in water and alcohol. He found that the first, third, and fourth assumptions were all perfectly satisfied in his experiment. Therefore, since our particles have a lower density and our liquid a higher viscosity, there should be no question of slipping.

(e) Fifth Assumption. The fifth assumption depends upon the velocity of fall and the viscosity. Arnold found that when the radii of his particles were less than $0.6 \frac{\eta}{V \sigma_m}$ this assumption was satisfactorily fulfilled. If we evaluate this we will obtain the size of the largest particles that should be present during the sedimentation.

$$r_{max.} = \frac{0.6\,\eta}{V \sigma_m}$$

$$\text{but } V = \frac{2g(\sigma - \sigma_m) r^2}{9\eta}$$

$$\therefore r_{max.} = \frac{5.4\,\eta^2}{2g\, r_{max.}^2\, \sigma_m\, (\sigma - \sigma_m)}$$

$$r^3_{max.} = \frac{5.4 \times .01814^2}{2 \times 980 \times 0.817\,(3.15 - .817)}$$

$$r = .0078\,\text{cm.}$$

$$\text{or dia.} = 156 \text{ microns.}$$

Since our largest diameter is 90 microns, there is a good margin of safety left.

44. Correction Factor for Deviation from Third Assumption. Thus it is seen that the assumptions are all satisfied with the exception of the third. The effect of this deviation can be

eliminated if an actual determination is made of the average diameter of the particles that just settle out from the top in a given time. If this diameter is compared with the diameter of the theoretical sphere of the same density which would settle out in the same time, a shape factor can be derived which can be used to correct the entire scale of sizes to be measured, provided the particles all have the same relative shape. A determination was made for cement as follows:
A small amount of material was placed in one of the sedimentation cylinders, mixed thoroughly, and allowed to settle for T minutes. The liquid with the particles still remaining in suspension was then withdrawn. The sediment on the bottom of the tube then contained all of the particles that were large enough to settle out from the top in time T or less, while the liquid contained only those that were smaller. The sediment was discarded while the liquid was placed in another tube and enough more liquid added to bring it up to level. It was then thoroughly stirred and allowed to settle for a $T + \Delta T$ and the liquid again removed. This time the sediment consisted of all the particles of the correct size to settle from the top between time T and $T + \Delta T$, together with a portion of the smaller particles that were below the top at the beginning of settling. The liquid contained only particles smaller than those corresponding to time $T + \Delta T$. These were discarded. Fresh liquid was added to the sediment and the stirring and settling for the time $T + \Delta t$ repeated. It is obvious that repetition eliminated a part of the material finer the $T + \Delta T$ limit, but that all of the particles

larger than those corresponding to this time, would always be found in the sediment. After a number of such repetitions, a small amount of material was obtained that would settle out, leaving the liquid clear in time $T + \Delta T$. Since all the particles that would settle out in the time T or less, had been eliminated in the first cut, the material remaining was nearly uniform in size, varying only between the size corresponding to times T and $T + \Delta T$. Microscopic slides were prepared and the statistical diameter of the particles was determined by measuring a large number of them with a microscope equipped with a filar eye piece.

Since the temperature, height, viscocity, and density of the sedimenting column were kept at all times at the standard values used in the regular operation of the machine, the material obtained should be exactly comparable to that settling out in the same time in the course of a regular analysis. Separation of four different sizes was effected and the statistical diameters determined, with results as shown;

Table I.

Sample	T	T+ΔT	Statistical Diameter Microns	Predicted Diameter Microns	Shape Factor
A	5'-15"	5'-30"	56.6	44.2	1.28
B	18'-0"	18'-45"	29.0	23.9	1.22
C	70'-0"	72'-0"	16.2	12.2	1.33
D	315'-0"	330'-0"	7.3	5.7	1.28

It is interesting to note the agreement between A and D, the largest and smallest fractions. Using this shape factor of I.28 the Stoke's Equation, becomes, when the values of viscosity and densities are inserted;

$$D = \frac{134}{\sqrt{T}}$$

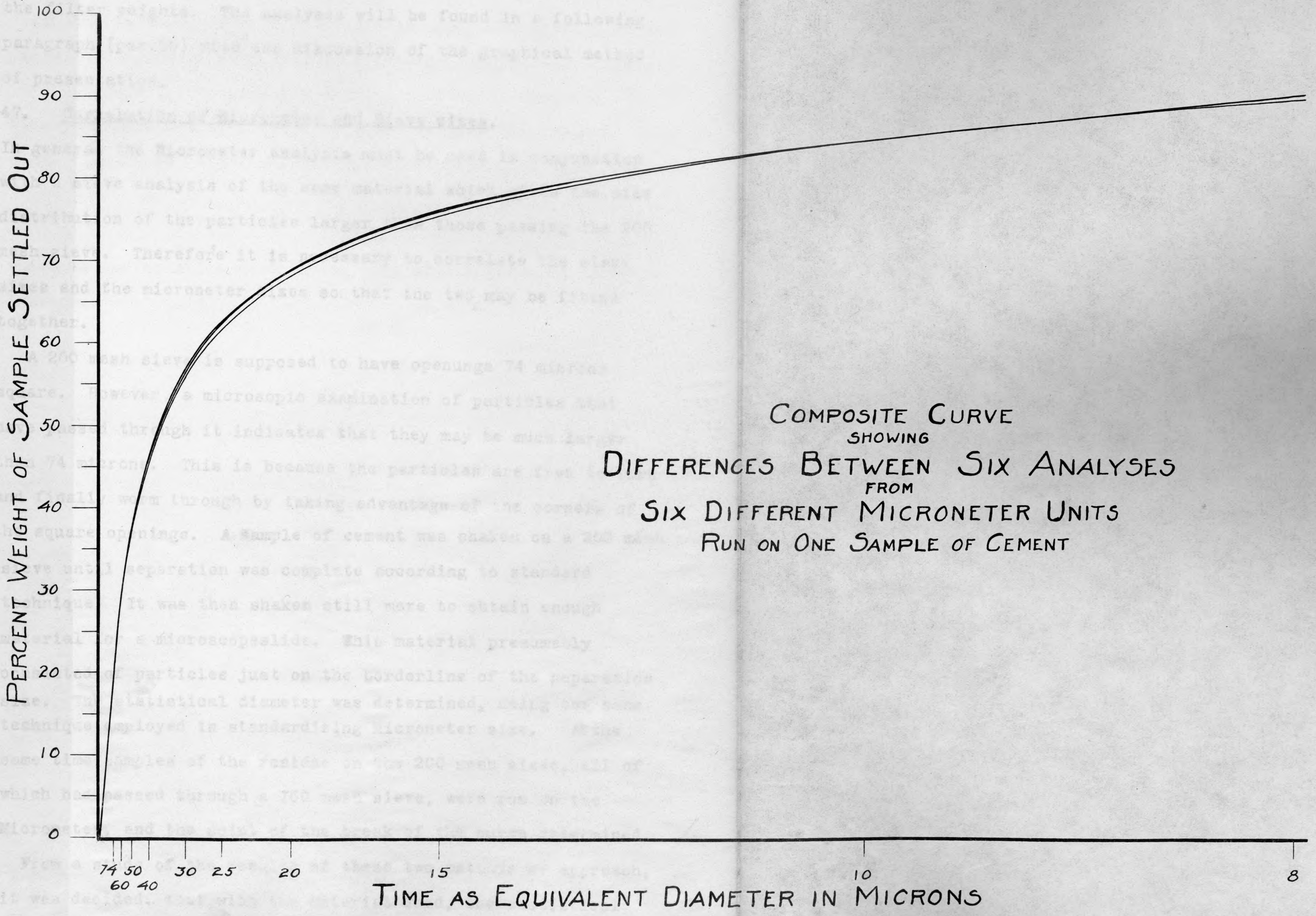
This work is good evidence that the relative shape of the particles remains the same over a large range of sizes.

45. Possibility of Interference Between Particles.

The validity of Stokes' equation for use with this apparatus may be questioned because of interference between particles during settling. The volume of the liquid is I900c.c. while the sample is never larger thanI6c.c. and is normally about Io c.c. It would seem therefore, that this objection is hardly valid. This was further corroborated by some check runs made with abnormally large samples. The curves obtained from the large samples were identical with those from normal samples of the same material.

46. Reproductibility of Results Obtained by the Microneter.

A check of the reproductibility of results, and correlations between the various units was desired,consequently a quantity of standard cement was divided into six samples and one run in each unit of the Microneter, some simultaneously, and some at another time. The plates obtained were projected in turn on a screen with an enlargement of about three diameters. The accompanying curve, Fig. (22) was obtained by tracing the upper edge of each record.



Composite Curve showing Differences Between Six Analyses from Six Different Microneter Units Run on One Sample of Cement

The differences shown indicate the overall deviations due both to difference in shape and in zero setting obtained from the filter weights. The analyses will be found in a following paragraph (par.50) with the discussion of the graphical method of presentation.

47. Correlation of Microneter and Sieve sizes.

In general the Microneter analysis must be used in conjunction with a sieve analysis of the same material which gives the size distribution of the particles larger than those passing the 200 mesh sieve. Therefore it is necessary to correlate the sieve sizes and the microneter sizes so that the two may be fitted together.

A 200 mesh sieve is supposed to have openungs 74 microns square. However, a microsopic examination of particles that have passed through it indicates that they may be much larger than 74 microns. This is because the particles are free to turn and finally worm through by taking advantage of the corners of the square openings. A sample of cement was shaken on a 200 mesh sieve until separation was complete according to standard technique. It was then shaken still more to obtain enough material for a microscopeslide. This material presumably consisted of particles just on the borderline of the separation size. The statistical diameter was determined, using the same technique employed in standardizing Microneter size. At the same time samples of the residue on the 200 mesh sieve, all of which had passed through a I50 mesh sieve, were run on the Microneter, and the point of the break of the curve determined.

From a study of the results of these two methods of approach, it was decided. that with the material used, the statistical diameter of particles just passing a 200 mesh sieve is 90 microns

Since sieve sizes are so well known and widely used, it was felt that it would be more desirable to apply the correction factor to the Microneter size to bring the two into agreement.

Therefore the statistical diameters obtained from the Microneter are all multiplied by 74/90 to give "Equivalent Sieve Diameters". This changes our Microneter size equation from

$$D = \frac{134}{\sqrt{T}} \quad \text{to} \quad D = \frac{110}{\sqrt{T}}$$

which is the equation used in the work of the grinding investigation.

Analysis of Plates.

48. Projection Apparatus For Plates.

The theoretical basis of the method used to obtain the size distribution from the sedimentation curve has already been discussed (par. I5, I6, I7). However nothing has been said about the technique of applying the method to the actual analysis of plates produced by the Microneter. In the first place, the plates used (Standard Lantern slides, 3 ¼" x 4") were rather small to be used directly. Therefore a small projector was constructrd to enlarge the curve sbout three diameters. A photograph of this is shown in Fig. (23).

Fig 23

The plate holder is arrangrd to have slow motion controls in both x and y directions, so that the projected image of the curve can be set in the correct position on the analysing screen. This screen is a piece of opal glass, ruled as indicated in Fig. 24

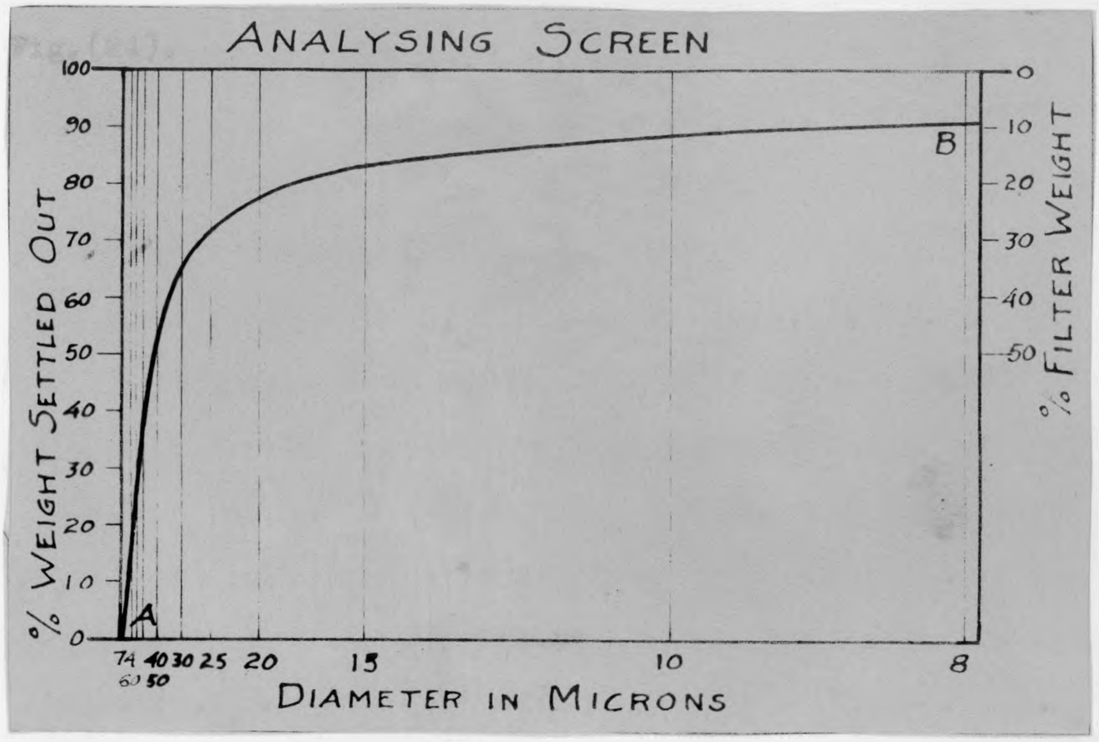


FIG. 24.

The scale on the y axis represents the 0/0 weight of the sample settled out, as indicated by the pressure. The axis is, of course, the time axis, but is graduated as size in microns, using the relation of the equation in (Par.45).

$$D = \frac{110}{\sqrt{T}}$$

The right hand ordinate scale is the one used in setting the vertical position of the curve by means of the weight of the material left in suspension at the end of the run, as determinrd by the filter weight.

The technique of analysis is as follows: A plate is placed in the projector and its image thrown on the screen, as in A-B Fig, (24).

The position is then adjusted until B comes at the proper value on the filter weight scale and A, or the curve produced, passes through the origin. The y axis intercepts of the tangents at the points of intersection of the curve and the micron lines are then obtained and recorded on the analysis sheet.

It is evident that if the intercepts and corresponding sizes, at the points of tangency, are recored, the results will be the familiar form of the cumulative weight vs. size table. A sample data sheet is shown in Fig. (25), which illustrates the method of analysis and calculation.

FORM R 555 2-29 2M

MICRONETER ANALYSIS SHEET

SAMPLE NO. =

% WEIGHT OF -200 MESH =

MICRONETER NO. =

SAMPLE DESCRIPTION =

FILTER WT. =

PLATE SETTING =

SAMPLE DENSITY =

J	K	L	M	N	P	R	S	T
EQUIVALENT SIEVE DIAMETER	CUMULATIVE % OF -200 MESH		CUMULATIVE % TOTAL SAMPLE	INDIVIDUAL % OF -200 MESH	% PER MICRON OF -200	% PER MICRON OF TOTAL SAMPLE	INDIVIDUAL SURFACE	
	INTERCEPT OF TANGENT	K × % WT. OF -200 MESH	L + (100 - % WT. OF -200 MESH)	ΔK	N / ΔJ	P × % WT. OF -200 MESH	FROM N AND NOMOGRAPH	
8								
10								
15								
20								
25								
30								
40								
50								
60								
74								

TOTAL SURFACE

DATE

UNIT

MICRONETER OPERATOR

PLATE ANALYSER

DATE ANALYZED

FIG. 25

49. Representation of Data.

After the size analysis has been made it is desirable to represent it graphically.

There are several ways of doing this. However, for our purpose there are two which are particularly adaptable, and will be the only ones described at present.

(A) Cumulative Percentage Curves

One of the simplest and the most interesting is the cumulative per-centage curve.

This can be plotted directly from the data as taken from the analysis without further calculation. It may be drawn with a cumulative percent weight as ordinate, and diameter as abscissa. However, if it is reversed and cumulative percent weight plotted as abscissa, and if the ordinate scale is plotted as the reciprocal of the diameter, it is found that the area under the curve represents the total surface of the sample. A specimen of such a curve is shown in Fig. (26)

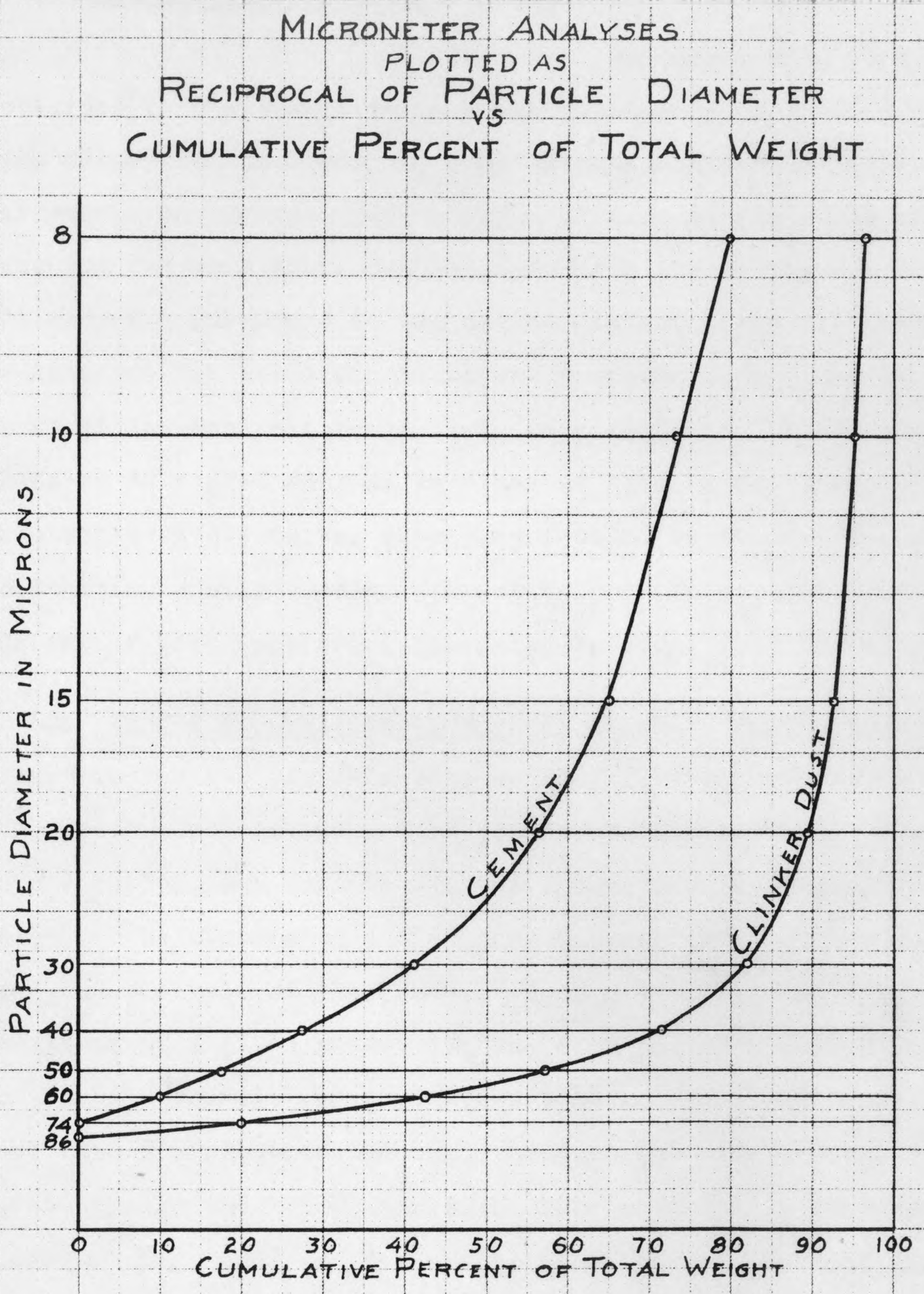

MICRONETER ANALYSES
PLOTTED AS
RECIPROCAL OF PARTICLE DIAMETER
VS
CUMULATIVE PERCENT OF TOTAL WEIGHT
8
10
15
20
30
40
50
60
74
86
PARTICLE DIAMETER IN MICRONS
CEMENT
CLINKER DUST
0
10
20
30
40
50
60
70
80
90
100
CUMULATIVE PERCENT OF TOTAL WEIGHT

FIG. 26

(B) Percent Per MIcron Curves (I)(I4)

Another useful curve is obtained if individual weights are plotted against the corresponding diameters. However, as many investigators have pointed out, erroneous conclusions may be drawn if care is not exercised in keeping the size increment the same over the entire range of diameters. Therefore it was decided to calculate all individual weights on the basis of one micron increments in diameter. Thus if the analysis shows that there is IO °/o of the sample between 30 and 40 microns in diameter this is represented as a rectangle on the curve, extending from 30 to 40 on the abscissa and having a height which represents one percent per micron. Curves of this type are illustrated by Figs. (27) to (29).

50. Curves Representing Graphical Check of Entire Analysis.

These curves are valuable because they give a graphical check on the performance of the entire procedure of determination of size distribution, and include all the errors due to sampling, operation of the Microneter, differences between units of the Microneter, and analysis of the plate. Each group represents six analyses of a given material, made from plates produced by each of the six units run at various times. Fig. (27) represents the size distribution the dust found in the clinker that would pass through the 200 mesh sieve. This material is comparatively coarse. Fig. (28) is from a commercial cement that contains a high percentage of fine material. These curves represent the analyses of the group shown in Fig. (22). Fig. (29) comes from a specially treated material containing a still higher percentage of fines.

It is safe to say that no better agreement would be found between similar duplicate samples of material, susceptible to sieve analysis, which had been sieved on six separate nests of sieves and the results plotted in a similar manner.

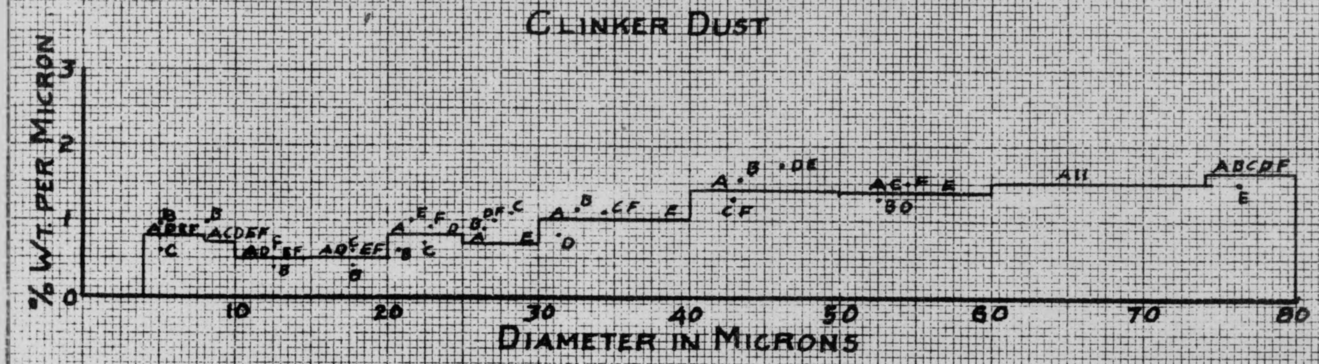


FIG. 27

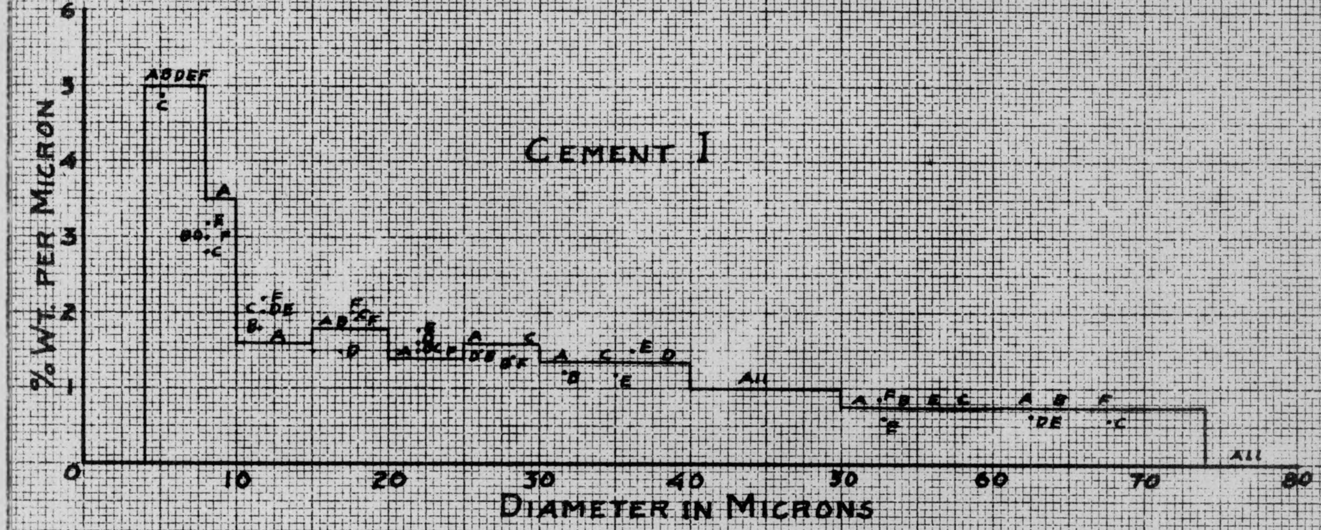


FIG. 28

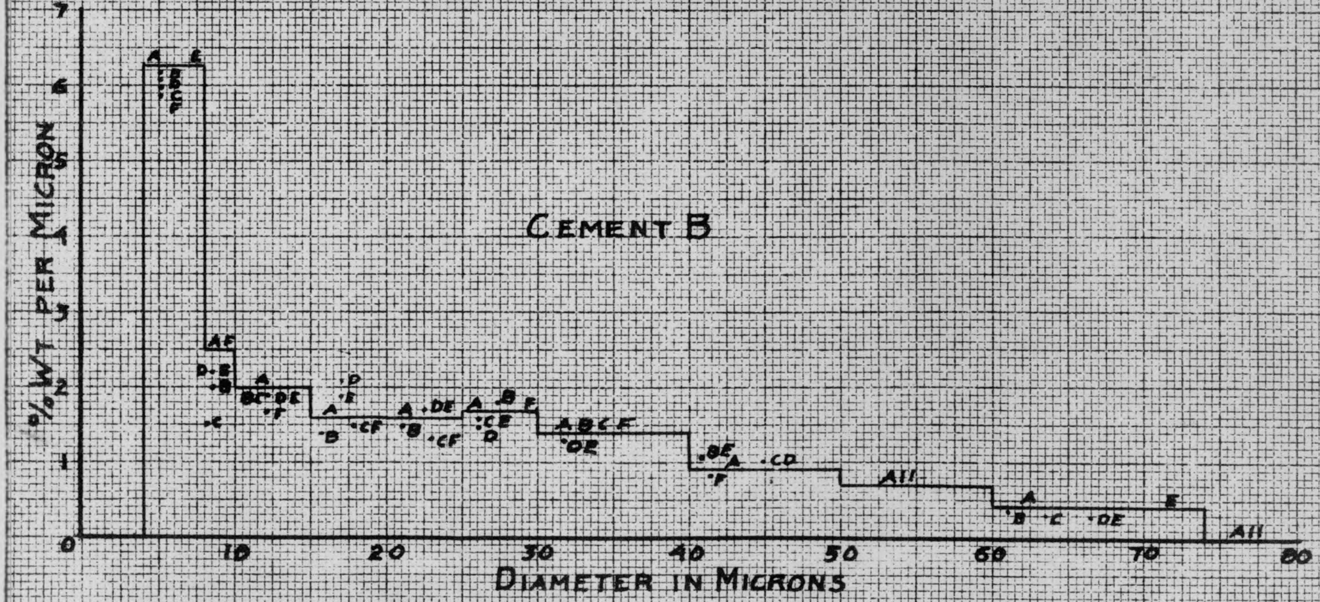


FIG. 29

PART II.

GRINDING STUDIES.

APPARATUS AND MATERIAL USED.

51. Scope of Problem. The general purpose of this investigation was stated in Part One (par. 6). It was to determine the effects of changes of the following variables upon the performance of a laboratory ball mill.

(1) Size of the particles making up the charge.
(2) Size of the balls used in the mill.
(3) Ratio of the weight of charge to the weight of balls.
(4) Time of grinding.

52. Laboratory Mill. The ball mill used for this work is shown in Fig. (30). It is a standard Abbe Jar Mill. Since the use of rather large steel balls was contemplated, it was felt that the standard porcelain jar probably would not have enough mechanical strength. Consequently the steel mill shown was constructed. The inside dimensions were made approximately equal to those of the porcelain jar. All of the work reported here was carried on in this steel mill. The length of the run was timed by means of the revolution counter fastened to the countershaft, as seen in the figure. The mill was driven by a constant speed motor, so runs of equal numbers of revolutions were also of equal duration. The inside diameter of the mill

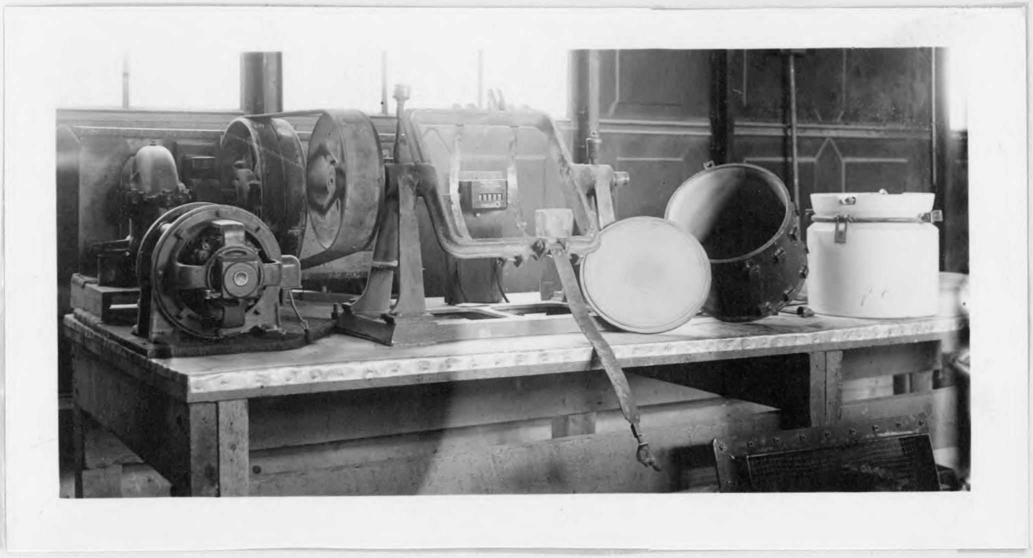


Fig. (30).

was 11" and the length 8 1/2 ". The mill was driven at 50.4 R.P.M. The countershift ran 38.1 R.P.M. Therefore when the revolution counter indicated 100 revolutions the mill had made 132 revolutions.

53. Balls Used for Grinding. The balls used in the mill were ball bearings. They were graded according to size and designated by the upper and lower limits of diameter to be found in a given lot. For example, -7/8" + 3/4" balls means that none of the balls were larger than 7/8" or smaller than 3/4" . The average diameter for each lot was determined from the density of the steel and the weight of a known number of balls. The various sizes of balls used are tabulated below. Enough of each size was available to furnish a full ball load for the mill.

TABLE II.

Ball Sizes For the Ball Mill.

Nom. Dia,	-1/2" + 3/8"	-5/8" + 1/2"	-3/4" + 5/8"	-7/8" + 3/4"	-1" + 7/8"	-1 1/8" + 1"	-1 1/4" + 1 1/8"	-1 3/8" + 1 1/4"	+ 1 3/8"
Ave. Dia.	0.441"	0.539"	0.653"	0.826"	0.916"	1.02"	1.14"	1.27"	1.49"

Density of steel in These balls = 7.848

The ball load was arbitrarily set at 21,000 g. and was kept at this value for all this work.

54. Material to be Ground. The material used in these investigations was chiefly Portland cement clinker. A small number of runs were made using Ottawa silica sand as the material to be ground.

55. Equipment Used to Determine the Size Distribution of the Material Being Ground. It was necessary to determine the size distribution of the particles of the charge material, both

before and after grinding. Between the limits of the maximum size and 74 microns (200 mesh sieve), a standard set of testing sieves was utilized. The Tyler series of sizes was used. The sieves were shaken mechanically by means of a Rotap. This equipment is seen in Fig. (31) together with a sample splitter or riffle, a small crusher, and a pulverizer. The last two were used very little in this investigation. Fig. (32) is one of the standard sieve sheets used. The Microneter was used to obtain the size distribution of particles smaller than 74 microns. A typical analysis sheet is shown in Fig. (35).

Fig. (31).

56. Selection of Charge Material for Tests. Cement material seems to vary considerably in it "grindability" or resistance to grinding. Variations are caused by age, chemical composition, and burning conditions in the kiln. Therefore it was necessary to exercise care in the selection of the clinker for use in the investigation, to ensure that a uniform material was secured. To accomplish this an attempt was made to obtain all of the material required for a complete series of runs at one time from one location in the clinker pile. When this was not possible the second lot was obtained from the same place as the first. Series E. and No.2 Time Runs were both long series and therefore more than one lot of clinker was required for each.

FORM R554 4-29 1M

(66 a)

SIEVE ANALYSIS SHEET

JAR MILL DATA

SAMPLE NO. =

SIEVE NEST NO. =

WT. OF SAMPLE =

CLINKER NO. =
" WT. =
" SIZE =
BALL SIZE =
" WT. =
NO. OF REVOLUTIONS =
DEGREE JAR COATING =
" BALL " =

SAMPLE DESCRIPTION

A	B	C	D	E	F	G	H
SIEVE MESH	SIZE OPENING MICRONS	CUMULATIVE WEIGHT	CUMULATIVE %	INDIVIDUAL %	% PER MICRON	INDIVIDUAL SURFACE	
		FROM BALANCE	$\frac{C}{\text{WT. OF SAMPLE}}$	ΔD	$\frac{E}{\Delta B}$	FROM E AND NOMOGRAPH	
3	6680						
4	4700						
6	3330						
10	1650						
14	1170						
20	833						
28	590						
35	417						
48	295						
65	208						
100	147						
150	104						
200	74						
-200							

TOTAL SURFACE

MILL OPERATOR

SAMPLE ANALYSER

DATE ANALYZED

FIG. 32

57. Apparatus for Sizing Charge Material. One of the main objectives of the investigation was to determine the effects caused by variations in the size of particles forming the charge. Hence it was necessary to separate the clinker into a number offractions,each of which consisted of particles whose diameters all fell within definite limits of size. It was feared that if a preliminary crusher was used to reduce the larger pieces to the desired size local strains and cracks would result,which would have the effect of causing the clinker to fracture more easily than it should at the beginning of grinding. in the mill. For this reason it was decided to screen the clinker as it came from the pile,thus avoiding any crushing or grinding until the charge was placed in the mill. The labor involved in hand screening the amount of material required for one of these series would have been prohibitive. Therefore a small set of vibrating screens was constructed. Fig.(33) shows a side view and Fig.(34) a front view of two stands of them. Each screen was provided with a bottom pan which carried all of the "throughput" down to the end and there dumped it at the top

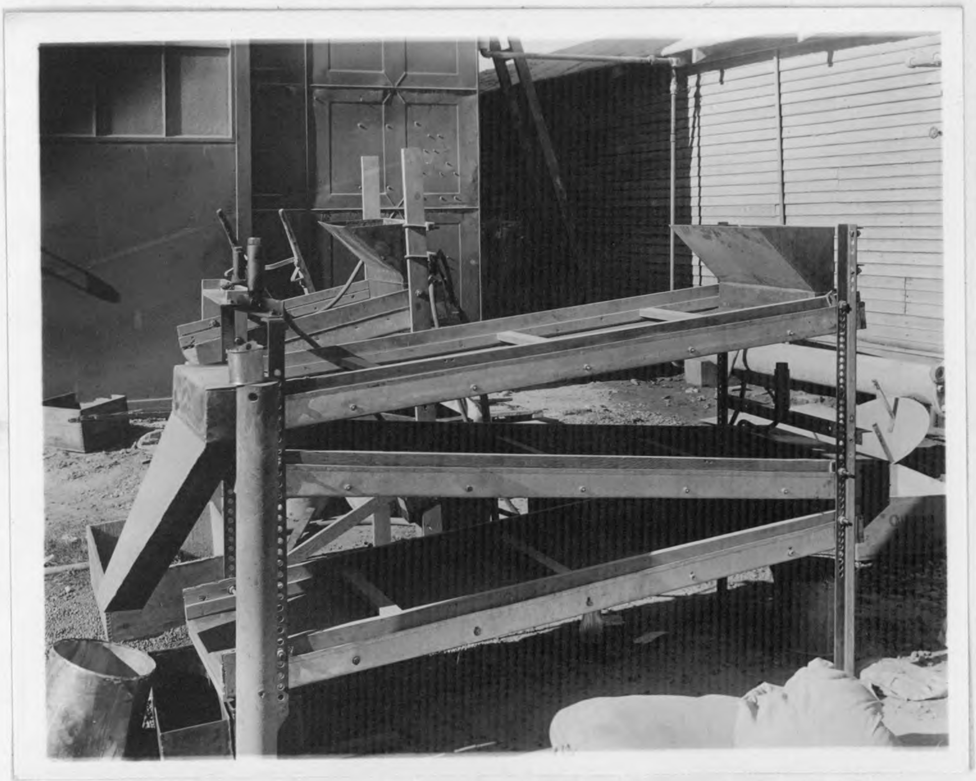


Fig.(33)

end of the screen below. An air vibrator was attached to each end of each stack. It was possible to obtain good separation with this apparatus.in a very much shorter time than by the use of hand screens.

Fig. (34).

58. Sizing, Mixing and Storing Material. The screens just described were chosen to have openings equal to those of the Tyler Standard series of sieves. The clinker fractions obtained were designated in the same manner as the balls, i.e., by giving the maximum and minimum diameter of particles to be found in the fraction. Sieve meshes were used to indicate the size instead of inches. Thus the charge described as "-20 + 28" signifies thst all of the material had passed through the 20 mesh screen and had been retainedon the 28 mesh. The clinker was separated into fractions with the following particle sizes.

Table III.

Designation	-3+6	-6+10	-10+14	-14 + 20	-20 + 28
Average Diameter in Microns	5004	2490	1410	1000	711
Designation	-28+35	-35+48	-48+65	-65+100	-100+200
Average Diameter in Microns	503	356	251	178	111

In addition, the -200 material was also segregated, but it had too wide a size distribution to assign an average diameter to it. After the pile of material had been carefully sieved, each fraction was thoroughly mixed, to secure uniformity throughout the fraction, and placed in cement sacks. When there was more than one sack full of a given fraction it was run through the riffle enough times to divide it into sack lots. This procedure made it certain that every mill charge from that fraction would have the same composition and properties. The sacks were all stored inside the laboratory until needed.

MILL PROCEDURE.

59. Mill Operation. When the details of a mill run had been decided, instructions were issued to the operator to proceed. This was done by giving him the sieve sheet to be used for that run. This sheet had been previously filled out to specify the charge identification number, its weight and particle size, the weight and ball size of the ball load, and the number of revolutions (as registered by the revolution counter). The operator then selected the specified material and weighed out the charge. The mill was inspected and cleaned if necessary to remove material from the last run. The correct ball load was selected and placed in the mill, then the charge was added, and the mill fastened in the frame. The mill was then started and allowed to run for the specified number of revolutions. At the end of that time it was removed from the frame, opened, and the charge separated from the balls by means of a coarse screen. The degree of ball and mill coating was recorded on the sieve sheet. The number of the run was placed on the sheet. The system used for this was very simple; it consisted

of a letter and three numbers. The letter indicated the mill on which the run had been made. The first two numbers indicated the month and the day, while the third gave the number of the run on that day. Thus J-2-21-9 indicated that the run was made on the jar mill on the 21st of February, and that it was the 9th run of the day,

60. Determination of the Size Distribution of the Ground Charge,

a. Sieves. After the ground charge had been separated from the balls it was passed through the riffle to obtain a representative sample. About 1000 grams were saved from each mill run. This was stored in quart cans, provided with tight fitting lids, and numbered to correspond to the run. Consequently there was always material to use for checking doubtful sieve or Micrometer analyses in case of need. Approximately 200 grams of the sample was riffled from the remainder of the charge, and the rest discarded. This was cut accurately to 200 grams at the balance, and then sieved for 15 minutes on the Rotap, as described in par. 55. It was finished by hand if the sample contained much flour. The fractions were then weighed on the balance to the nearest 0.5 gram. The smallest size was weighed first, then the next added to it, and so on. The weights were recorded in column C, or Cumulative Weight column, on the sieve sheet. There was one deviation from the above procedure. The -200 material was weighed separately, placed in a small tin can, and sent to the Microneter. If not enough -200 material was obtained from the first sieving it was repeated with a second 200 gram sample, or as many more as necessary.

b. Microneter. When it was desired to have the Microneter complete the analysis the same procedure was followed as has been outlined in reference to the mill operation (Par.59). The Microneter AnalysisSheet for that sample was given the operator, with the sample number, description, and percent weight of the -200 mesh fraction filled in by theman in charge of the laboratory. The analysis was made as described in Part I. (par. 40,41,42and 48).

FORM R554 1-23 1M

SIEVE ANALYSIS SHEET

JAR MILL DATA

SAMPLE NO. = J-3-4-5

SIEVE NEST NO. = 2

WT. OF SAMPLE = 200 G.

CLINKER NO. = C-2-19-1
" WT. = 1600 G
" SIZE = −20 + 28
BALL SIZE = $-\frac{7}{8}'' + \frac{3}{4}''$
" WT. = 21,000 G.
NO. OF REVOLUTIONS = 600
DEGREE JAR COATING = NONE
" BALL " = "

SAMPLE DESCRIPTION
SERIES E

A	B	C	D	E	F	G	H
SIEVE MESH	SIZE OPENING MICRONS	CUMULATIVE WEIGHT	CUMULATIVE %	INDIVIDUAL %	% PER MICRON	INDIVIDUAL SURFACE	NUMBER × 10^{-3}
		FROM BALANCE	$\frac{C}{\text{WT. OF SAMPLE}}$	ΔD	$\frac{E}{\Delta B}$	FROM E AND NOMOGRAPH	FROM E AND NOMOGRAPH
3	6680						
4	4700						
6	3330						
10	1650						
14	1170						
20	833	0	0	0	0		
28	590	3	1.5	1.5	0.006	42	2.6
35	417	12	6.0	4.5	0.025	174	21.5
48	295	28	14.0	8.	0.065	450	120
65	208	50	25	11	0.126	860	430
100	147	76	38	13	0.213	1420	1400
150	104	104	52	14	0.325	2200	4150
200	74	124	62	10	0.333	2200	8600
−200		200	100	38			

TOTAL SURFACE 7346

MILL OPERATOR H.K.

SAMPLE ANALYSER H.K.

DATE ANALYZED 3-4-29

FIG. 35

FORM R 555 2-29 2M

MICRONETER ANALYSIS SHEET

SAMPLE NO. = J-3-4-5
MICRONETER NO. = 1243
FILTER WT. = 2.56 G.
PLATE SETTING = 8
SAMPLE DENSITY = 3.11

% WEIGHT OF -200 MESH = 38
SAMPLE DESCRIPTION = SERIES E
1600 G. OF C-2-19-1 SIZED -20+28
21000 G. OF $-\frac{7}{8}" + \frac{3}{4}"$ BALLS
600 REV.

J	K	L	M	N	P	R	S	T
EQUIVALENT SIEVE DIAMETER	CUMULATIVE % OF -200 MESH		CUMULATIVE % TOTAL SAMPLE	INDIVIDUAL % OF -200 MESH	% PER MICRON OF -200	% PER MICRON OF TOTAL SAMPLE	INDIVIDUAL SURFACE	NUMBER $\times 10^{-6}$
	INTERCEPT OF TANGENT	K × % WT. OF -200 MESH	L + (100—% WT. OF —200 MESH)	Δ K	$\frac{N}{\Delta J}$	P × % WT. OF -200 MESH	FROM N AND NOMOGRAPH	FROM N AND NOMOGRAPH
-8	100	38	100	12.0	3.0	1.14	38,500	35,000
8	88	33.5	95.5	5.0	2.5	0.95	11,000	4,400
10	83	31.5	93.5	9.5	1.9	0.72	15,000	3,100
15	73.5	28	90.0	7.5	1.5	0.57	8,200	900
20	66	25	87	8.0	1.6	0.61	6,900	440
25	58	22	84	8.0	1.6	0.61	5,600	230
30	50	19	81	14.5	1.45	0.55	8,000	210
40	35.5	13.5	75.5	12.0	1.2	0.46	5,200	81
50	23.5	9	71.	10.5	1.05	0.40	3,700	39
60	13.	5	67	13.0	0.93	0.35	3,900	28
74	0	0	62	0	0	0	0	0

TOTAL SURFACE	106,000
-200 SURFACE OF TOTAL SAMPLE	40,200
+200 SURFACE	7,350
TOTAL	47,550
ORIGINAL SURFACE	2,750
SURFACE PRODUCED	44,800

DATE 3-15-29
UNIT II
MICRONETER OPERATOR H.H.

PLATE ANALYSER F.W.

DATE ANALYZED 3-20-29

FIG. 36

c. Computation. The columns C and K, in the Sieve and Micrometer Sheets respectively, represented the observed data. The other columns were filled in by use of the relations shown at the head of the column. H. and T were used for the Number of Particles. These were obtained from the same settings on the Nomograph as columns G and S. Columns G, H, S, and T were all based on a 100 gram total sample. A computation of the total surface produced in the grinding was made at the bottom of the column S. The sum of the individual terms of S gave the surface per 100 grams of the -200 mesh material in the ground charge, The sum of the column G gave the surface of the +200 fraction in each 100 grams of the whole charge. Therefore when this was added to the above value the result was the total surface per 100 grams of ground charge. The initial surface of the material before grinding was then subtracted, leaving the surface produced per 100 grams of charge. Fig.(35) is a completed Sieve Sheet for one of the runs of Series E and Fig.(36) is the corresponding Micrometer Sheet.

DESCRIPTION OF SERIES OF RUNS.

61. Series E. The first of the two sets of runs that are to be discussed is Series E. This group was designed primarily to furnish data on effects upon the grinding action in the mill of changes in ball size, charge particle size, and charge weight. The ball load was kept constant, throughout the series at 21000 grams. The time of grinding was also held constant at 600 revolutions of the countershaft (i.e. 782 revolutions of the mill.) Table 4 presents the schedule of this series.

TABLE 4

Series E

Ball Size	CHARGE WEIGHT						
	1600 g.	2000g.	2400g.	2800g.	3200g.	3600g.	4000g.
	CHARGE PARTICLE SIZE						
$-\frac{1}{2}''+\frac{3}{8}''$	-14+20 -28+35 -48+65	-28+35	-28+35	-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200	-28+35	-28+35	-14+20 -28+35 -48+65
$-\frac{5}{8}''+\frac{1}{2}''$				-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200			
$-\frac{3}{4}''+\frac{5}{8}''$	-28+35	-28+35	-28+35	-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200	-28+35	-28+35	-28+35

TABLE 4

Series E (Cont'd)

Ball Size	CHARGE WEIGHT						
	1600g	2000g	2400g	2800g	3200g	3600g	4000g
	CHARGE PARTICLE SIZE						
$-\frac{7}{8}''+\frac{3}{4}''$	-10+14 -20+28 -35+48	-20+28	-20+28	-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200	-20+28	-20+28	-10+14 -20+28 -35+48
$-1''+\frac{7}{8}''$				-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200			
$-1\frac{1}{8}''+1''$	-6+10 -20+28	-20+28	-20+28	-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200	-20+28	-20+28	-6+10 -20+28

TABLE 4

Series E (Concluded)

	CHARGE WEIGHT						
Ball Size	1600g	2000g	2400g	2800g	3200g	3600g	4000g
	CHARGE PARTICLE SIZE						
-1 $\frac{1}{4}$"+1 $\frac{1}{8}$"				-6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200			
-1 $\frac{3}{8}$"+1 $\frac{1}{4}$"	-6+10 -20+28 -35+48	-20+28	-20+28	-3+6 -6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200	-20+28	-20+28	-6+10 -20+28 -35+48
+1 $\frac{3}{8}$"	-10+14			-3+6 -6+10 -10+14 -14+20 -20+28 -28+35 -35+48 -48+65 -65+100 -100+200 -200			-10+14

The 2800 gram charge weight was taken as the base because, on the average, it had the correct volume to just fill the voids between the balls. Consequently, in the three groups of runs with lighter charges, the material did not completely fill the voids while, on the other hand, there was an excess of charge over that required for this purpose in the three groups with the heavier charges. The runs with the 2800 gram charges were made to ascertain the effects of variations in ball and particle sizes; those made with constant particle size to study variations of charge weight. The other runs, at maximum and minimum charge weights, were for the purpose of providing secondary constant charge weight groups to indicate whether the grinding action was the same at the two extremes as it was with the base charge weight. The complete series consisted of 168 runs.

62. No. 2 Time Series Runs. The second major group was called No.2 Time Series. Its chief objective was to furnish evidence of the effects of length of time grinding. The data obtained was very useful for supplementing that from Series E. Thisgroup also contained a limited number of runs made with silica sand instead of clinker. All of the runs were with the standard ball load of 21,000 g. Table 5 shows the plan of this series. In this table every possible combination of factors in each horizontal group represents a run. For example, the third group contains 105 runs; seven runs of different lengths for five charges of varying particle sizes, each of which was run with three different sizes of balls. The number of runs represented in the whole table is 148. The 2800 gram charge was again taken as the base, and the widest variations in the three factors were made with this charge weight. The runs whose duration was 60 revolutions served as direct checks on the similar ones in Series E.

TABLE 5

Number 2 - TIME EFFECT RUNS

Material Used	Ball Size	Charge Weight	Charge Particle Size	Duration of Run
Clinker	-1 1/8"+1"	2000 g	-14+20 --48+65	300 Rev. 600 " 900 " 1200 " 1500 " 2100 " 3000 "
Silica Sand	-1 1/8"+1"	2000 g	-20+28	300 " 600 " 900 " 1200 " 1500 " 2100 " 3000 " 6000 "
Clinker	-1/2"+3/8" -7/8"+3/4" -1 3/8"+1 1/4"	2800 g	-6+10 -14+20 -28+35 -48+65 -100+200	300 " 600 " 900 " 1200 " 1500 " 2100 " 3000 "
Clinker	-7/8"+3/4"	4000 g	-10+14 -28+35 -65+100	300 " 600 " 900 " 1200 " 1500 " 2100 " 3000 "

63. Presentation of Data. Obviously it would be impossible to present all of the original data obtained from these two series of runs without making this treatise very bulky. Therefore the material needed will be presented as each separate item of the results is discussed. The presentation will be made either in the form of tables or charts. In general, when the values shown are functions of the original data, instead of the data itself, the calculations involved were very simple and should not introduce any uncertainty into the results.

RESULTS AND CONCLUSIONS.

I. Effects of Variation on Length of Time of Grinding.

64. Statement of Rittenger's Law. For many years there has been much controversy about the energy relations involved in a grinding process. Rittenger(17) (19) (20) stated that the energy required was proportional to the new surface produced. Kick(18) (21) maintained that equal amounts of energy were required to produce equal diameter reduction ratios. Stated mathematically, these become

Rittenger's Law Energy $= C(S_2 - S_1)$

Kick's Law " $= B \log \frac{D_1}{D_2}$

where S_1 initial surface D_1 = initial diameter
S_2 final " D_2 = final " "
B and C are constants

For equal weights of material, the surface is proportional to the reciprocal of the diameter of the particles. Therefore surfaces may be substituted for the diameters in the above expressions, or vice versa.

$$D_1 = \frac{a}{S_1} \quad D_2 = \frac{a}{S_2}$$

Rittenger's Law Energy $= C(S_2 - S_1) = C'\left(\frac{1}{D_2} - \frac{1}{D_1}\right)$

Kick's Law Energy $= B \log \frac{D_1}{D_2} = B \log \frac{S_2}{S_1}$

It is obvious that both of these laws cannot be true, for they differ greatly. For an example, assume that it requires an amount of energy E to reduce a unit weight of a given material from a diameter of 1" to ½". If the material was to be reduced from 1" to 1/10" instead, Kick's Law would predict that the energy required would be

$$E' = E \frac{\log \frac{1}{\frac{1}{10}}}{\log \frac{1}{\frac{1}{2}}} = E \frac{\log 10}{\log 2} = 3.32 E$$

While

from Rittenger's Law

$$E' = E \frac{\left(\frac{1}{\frac{1}{10}} - \frac{1}{1}\right)}{\left(\frac{1}{\frac{1}{2}} - \frac{1}{1}\right)} = E \frac{(10-1)}{(2-1)} = 9E$$

One of the reasons the dispute over these two laws has not been settled is the lack of knowledge of the complete size distribution of the product. Without this knowledge it has been impossible to determine just what grinding had been done in a given test

65. Data From This Investigation. The present investigation has some evidence to offer on this question. No.2 Time Series was especially designed to furnish information about the effects of varying the time of grinding. In a ball or tube mill, the energy input depends primarily upon the speed and the ball load. It is only slightly affected by the weight of charge (within limits) and not at all by the particle size. Therefore,when speed, ball load,and charge weight are kept constant, the energy input is directly proportional to thelength of time of grinding. No.2 Time Series contains, as reference to Table 5 shows, a number of series of runs of varying lengths, in which the ballmsize, ball load,charge weight, and initial particle diameter were maintained constant throughout the series. The surface produced in

each run has been plotted against the corresponding length in of the run for each series. If the energy required is proportional to the surface produced (i.e. Rittenger's Law) these points should fall in a straight line, while if Kick's Law is true, the points will beon a logarithmic curve. Figs. (37) to (42) are the curves plotted from this data.

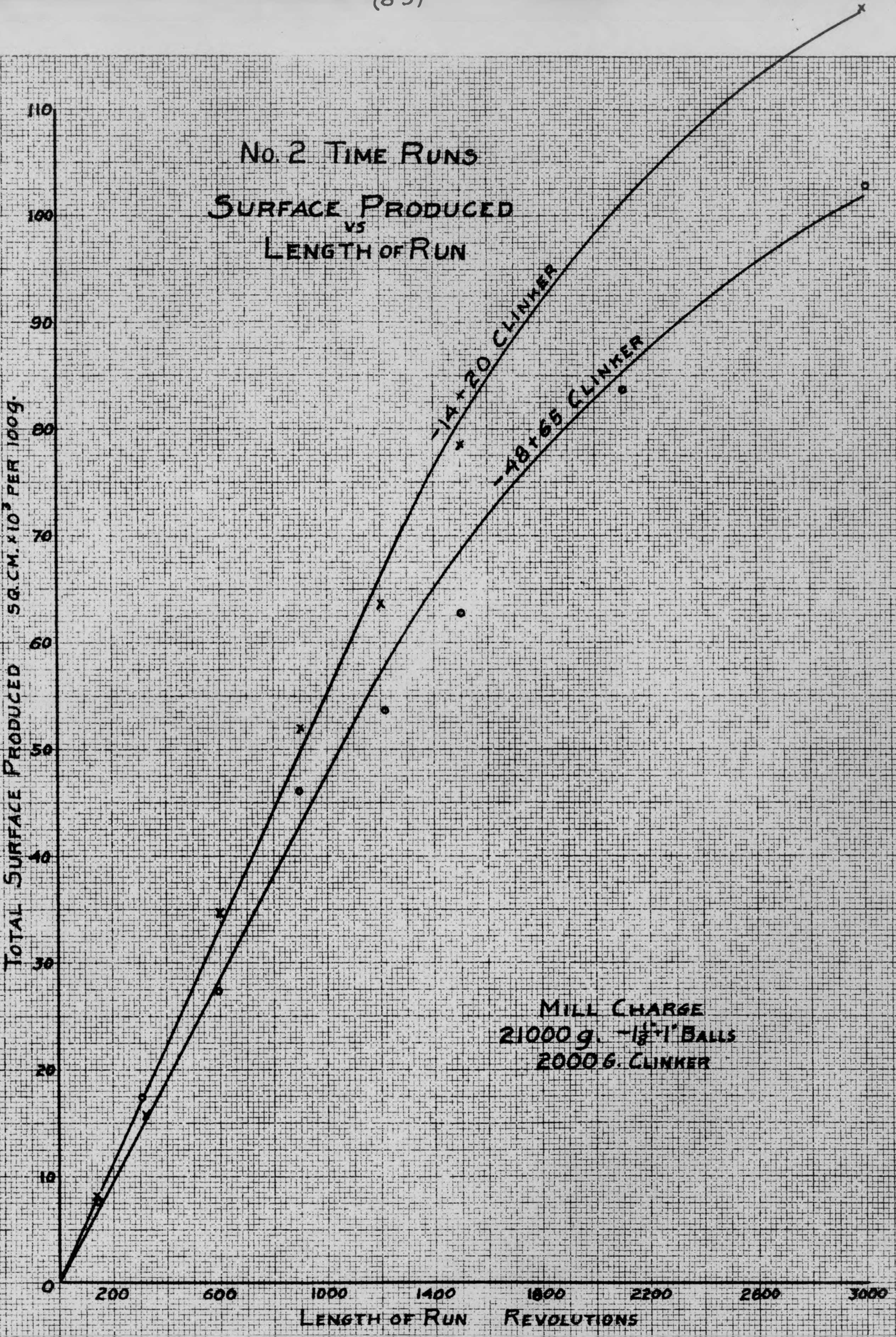
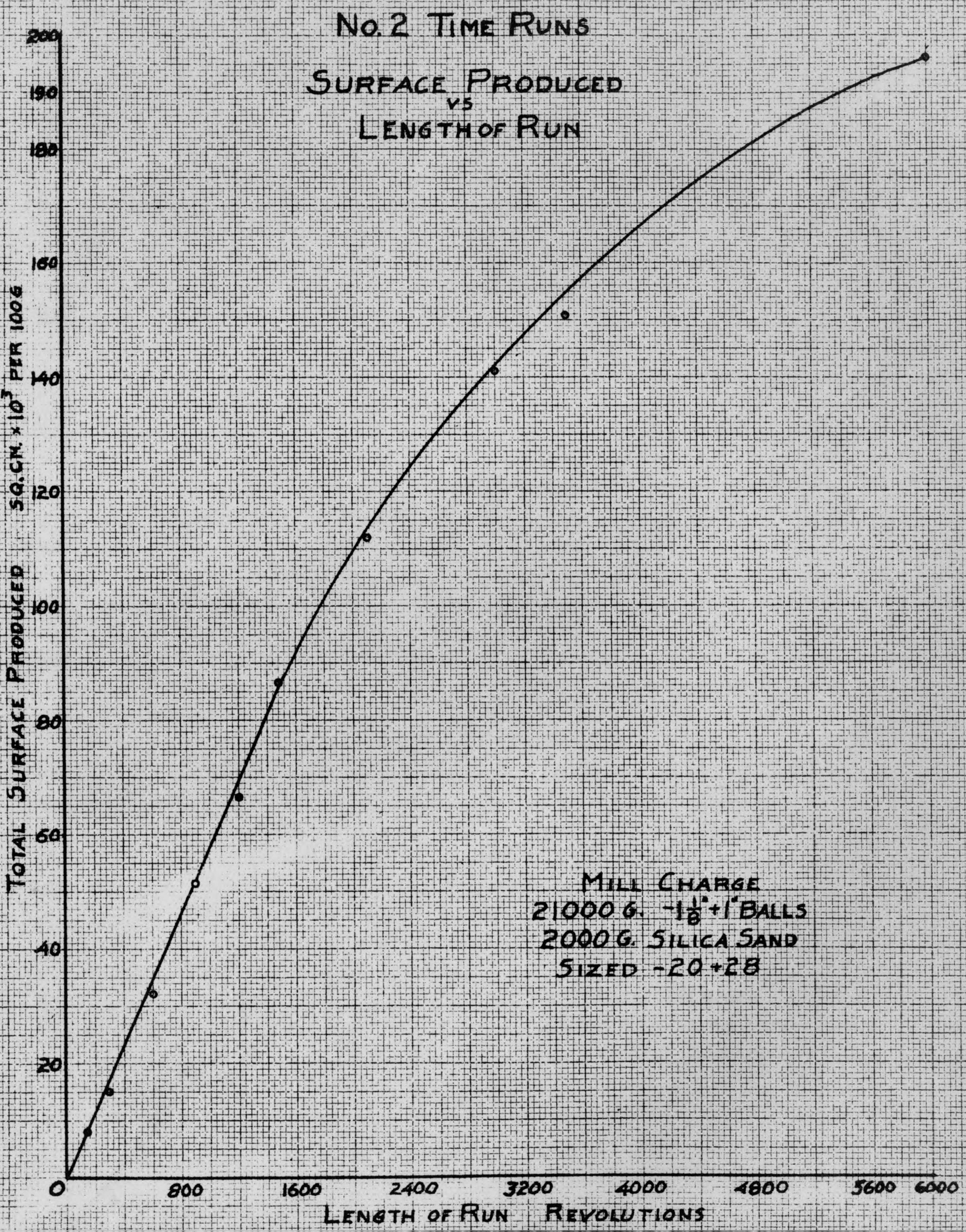

No. 2 Time Runs
Surface Produced vs Length of Run
-14+20 Clinker
-48+65 Clinker
Mill Charge
21000 g. -1½"-1" Balls
2000 G. Clinker
Total Surface Produced sq.cm. × 10³ per 100g.
0
10
20
30
40
50
60
70
80
90
100
110
200
600
1000
1400
1800
2200
2600
3000
Length of Run Revolutions

Fig. 37



NO. 2 TIME RUNS

SURFACE PRODUCED
VS
LENGTH OF RUN

MILL CHARGE
21000 G. $-1\frac{1}{8}'' + 1''$ BALLS
2000 G. SILICA SAND
SIZED -20+28

FIG. 38

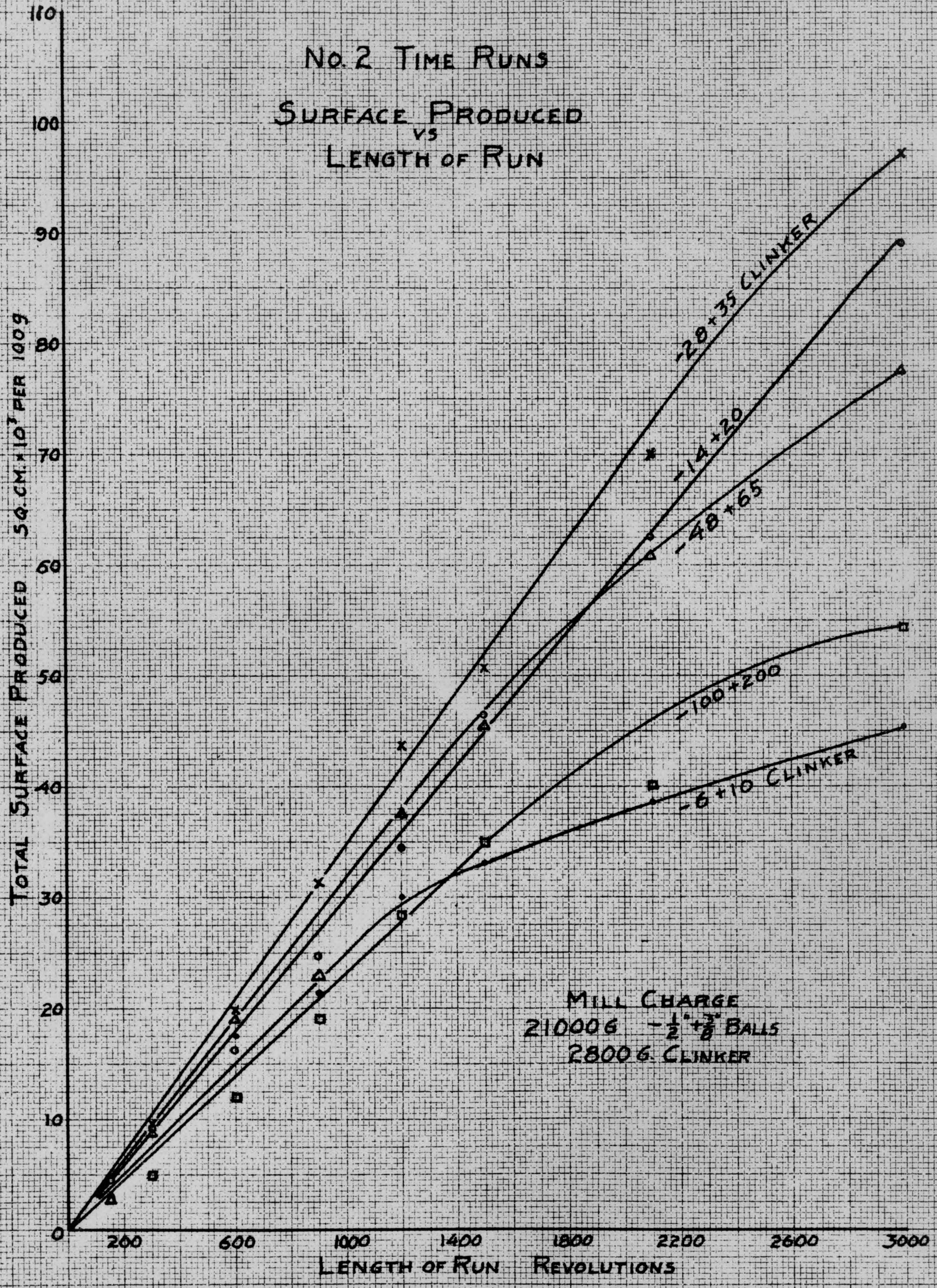

No. 2 Time Runs
Surface Produced vs Length of Run
Total Surface Produced Sq. Cm. × 10³ per 100g
Length of Run Revolutions
0
10
20
30
40
50
60
70
80
90
100
110
200
600
1000
1400
1800
2200
2600
3000
-28+35 Clinker
-14+20
-48+65
-100+200
-6+10 Clinker
Mill Charge
21000G -½"+⅜" Balls
2800G. Clinker

Fig. 39

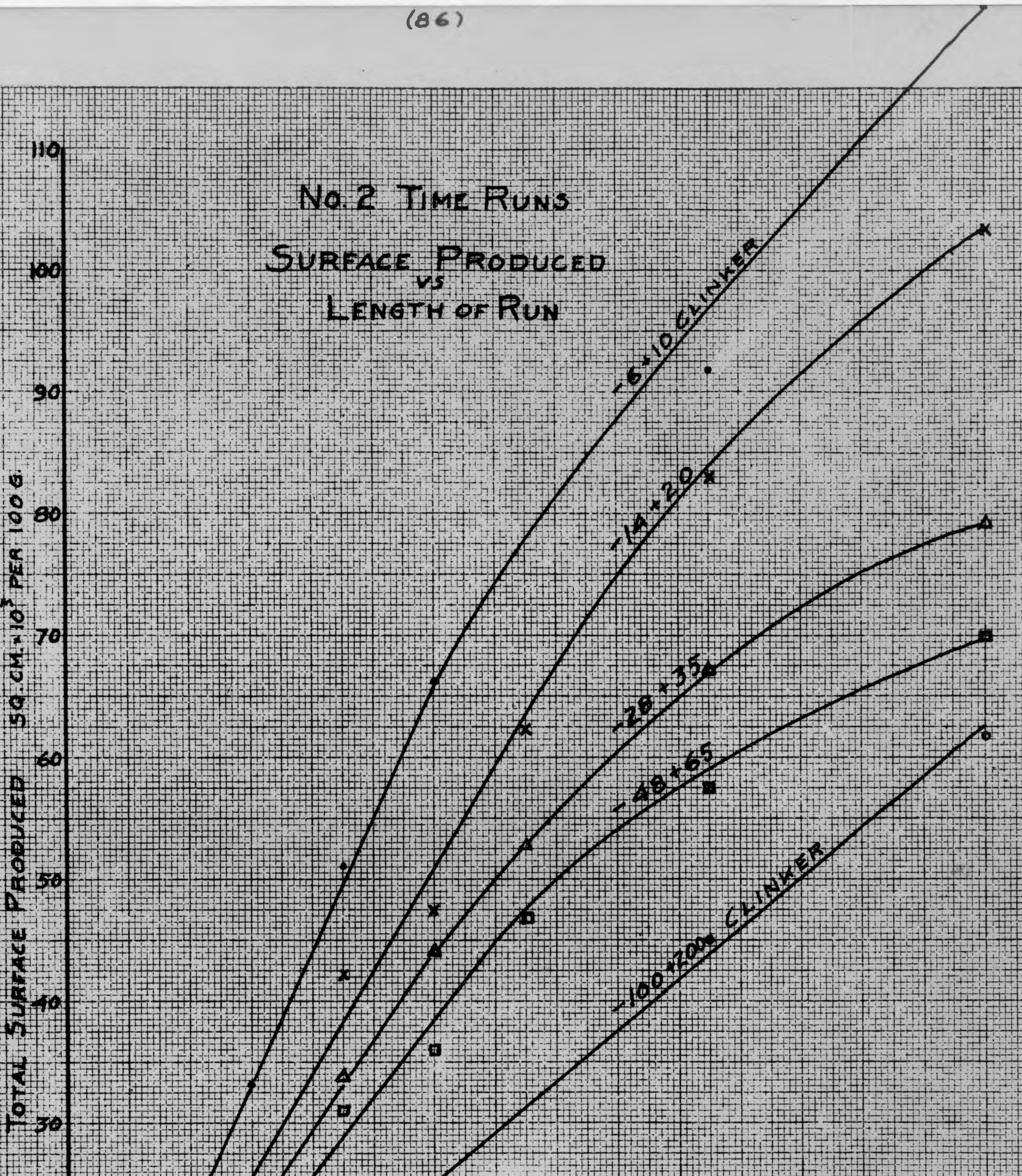
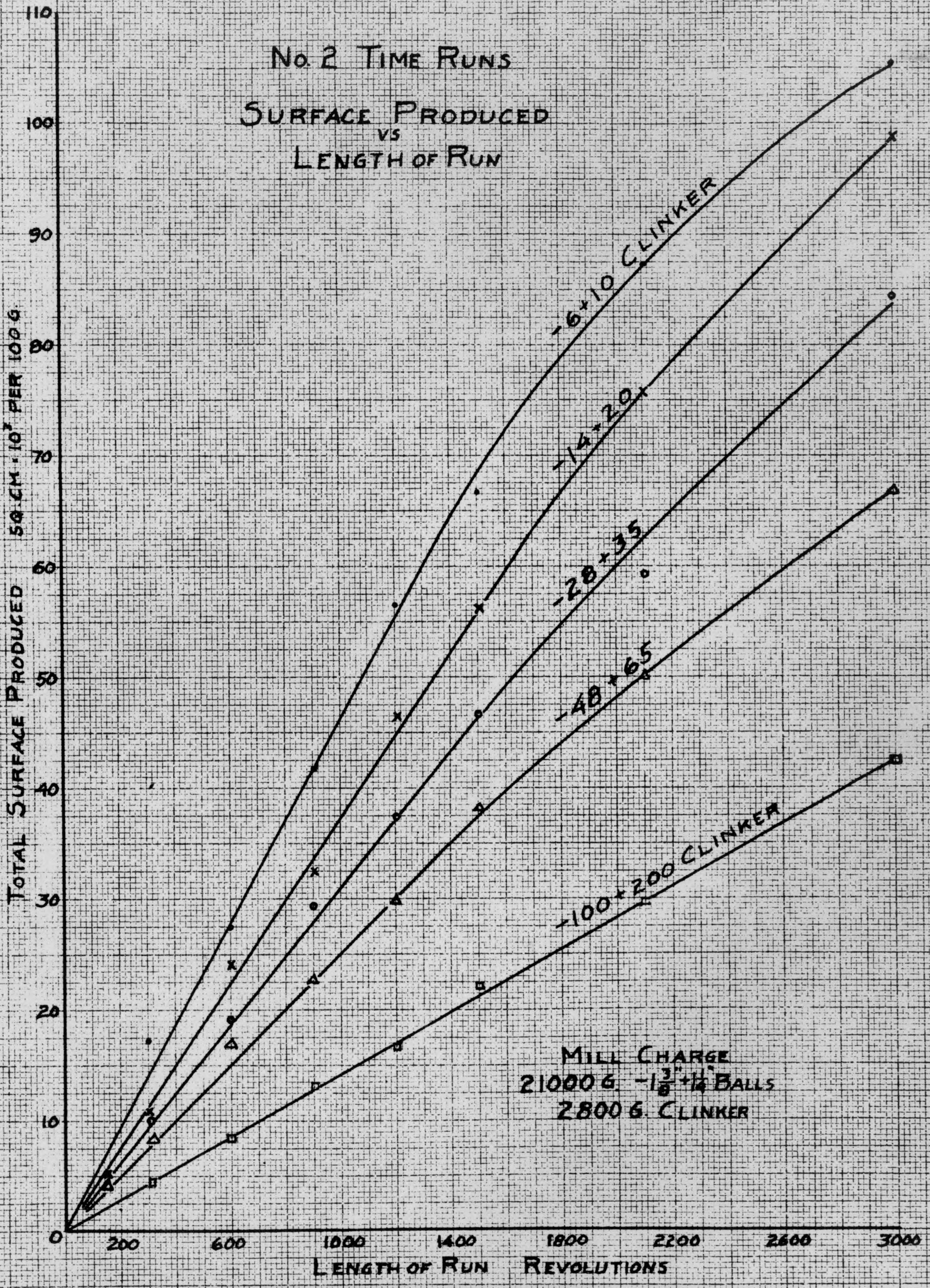

No. 2 Time Runs
Surface Produced vs Length of Run
Total Surface Produced sq.cm. × 10³ per 100 g.
Length of Run Revolutions
0
10
20
30
40
50
60
70
80
90
100
110
200
600
1000
1400
1800
2200
2600
3000
-6+10 Clinker
-14+20
-28+35
-48+65
-100+200 Clinker
Mill Charge
21000 g. -7"/8 + 3"/4 Balls
2800 g. Clinker

Fig. 40



No. 2 Time Runs

Surface Produced vs Length of Run

Mill Charge
21000 G. $-1\frac{3}{8}"+1\frac{1}{4}"$ Balls
2800 G. Clinker

FIG. 41

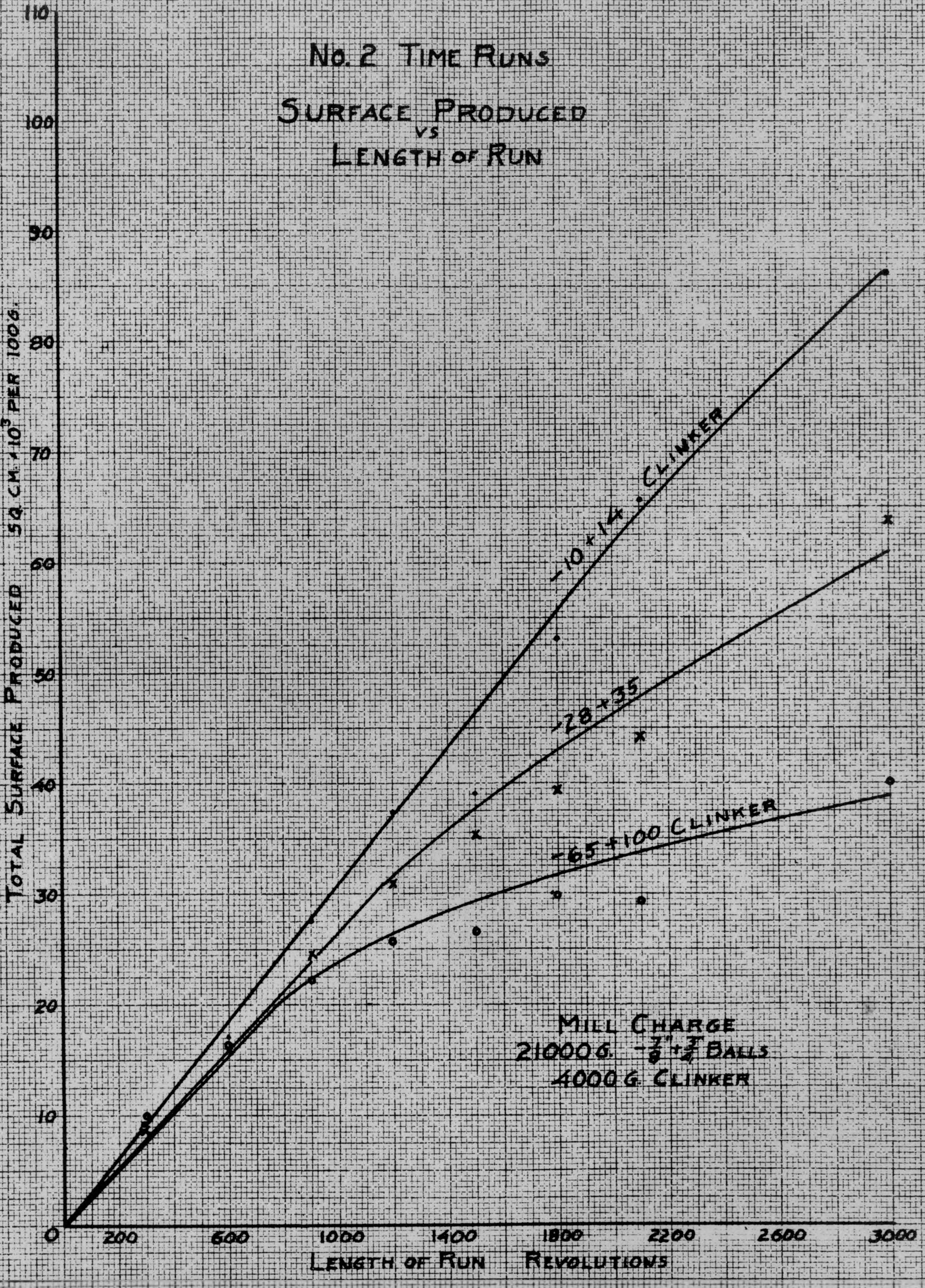

No. 2 Time Runs
Surface Produced vs Length of Run
Total Surface Produced sq. cm. × 10³ per 100 G.
Length of Run Revolutions
-10 +14 Clinker
-28 +35
-65 +100 Clinker
Mill Charge
21000 G. -7/8" +3/4 Balls
4000 G. Clinker
0
10
20
30
40
50
60
70
80
90
100
110
200
600
1000
1400
1800
2200
2600
3000

Fig. 42

66. Discussion of Data. Several facts are brought out by these curves. First, there is no evidence to show that Kick's Law holds over any part of the range covered by these experiments. Second, Rittenger's Lawn holds accurately for moderate amounts of grinding in all of the cases studied. Third, the deviations from Rittenger's Law shown after long periods of grinding are, without exception, in the direction of still greater divergence from Kick's Law. In other words,after long periods of grinding the surface produced in any given number of revolutions is less than it was initially. Fourth, the constant of proportionality C in Rittenger's Law, is a function of the ball and particle diameters. Fifth, equal amounts of grinding produce approximately the same amounts of surface when either silica sand or clinker is the substance ground. This should not be given too much weight, because, although silica sand may be a uniform substance, clinker is not. Hence for another kind of clinker this relation might not hold. Sixth, the deviation from Rittenger's Law shown by the longer runs may be partially accounted for by the tendency of the very finely ground charge to stick to the balls and the sides of the mill. This "cushions" the balls as they cascade. The "ball coating" was only observed in the runs oflong duration. Seventh, point four offers another practical explanation of this deviation, in that, as theaverage particle size changes, the surface produced for equal amounts of grinding likewise changes; and, in general, it decreases with decreasing particle size.

67. Conclusions.

(1) Kick's Law does not apply to grinding in ball or tube mills.

(2) Rittenger's Law gives a good approximation of the

energy required to produce a given amount of surface.

(3) The energy required to produce a given amount of surface depends not only upon the physical properties of the material to be ground, but also upon the ratio of the diameters of the balls and the particles.

II. Effect of Changing the Ratio of Charge Weight to the Ball Load.

68. General Discussion. The statement is often made(19) that grinding processes are very inefficient, i.e., that the ratio $\frac{\text{Energy required for grinding}}{\text{Energy supplied to the mill}}$ is very small. It would be very desirable to increase this if possible. One of the variables that would be expected to affect the efficiency of a ball mill is the amount of charge present in the mill. Consequently, groups of runs were included in Series E for the purpose of obtaining data upon the effects of varying the amounts of charge with other conditions constant. The scope of these runs may be seen by referring to Table 4. They are the five horizontal groups of runs with constant initial particle size.

69. Data Obtained. The results of these runs are shown graphically by Figs. (43) and (44). The surface produced has been chosen as the measure of the energy required, because it was shown (Par. 66, 67) that Rittenger's Law is true for the conditions prevailing during these tests. It should be observed that, in this series of runs, the total surface produced must be used to measure energy required instead of the surface per 100 grams. The reason for this is that, unlike runs furnishing the data for Fig. (37) - (42), this group did not have the same charge weight, and therefore the surface produced in a given weight is not a measure of the increase in total surface. Accordingly,

the values for the ordinates, which represent the energy required, were obtained by multiplying the surface produced per 100 grams by the charge weight in hundreds of grams. The ordinate is also a measure of grinding efficiency because the speed of the will and the ball load, andtherefore the energy input, were left constant for all runs.

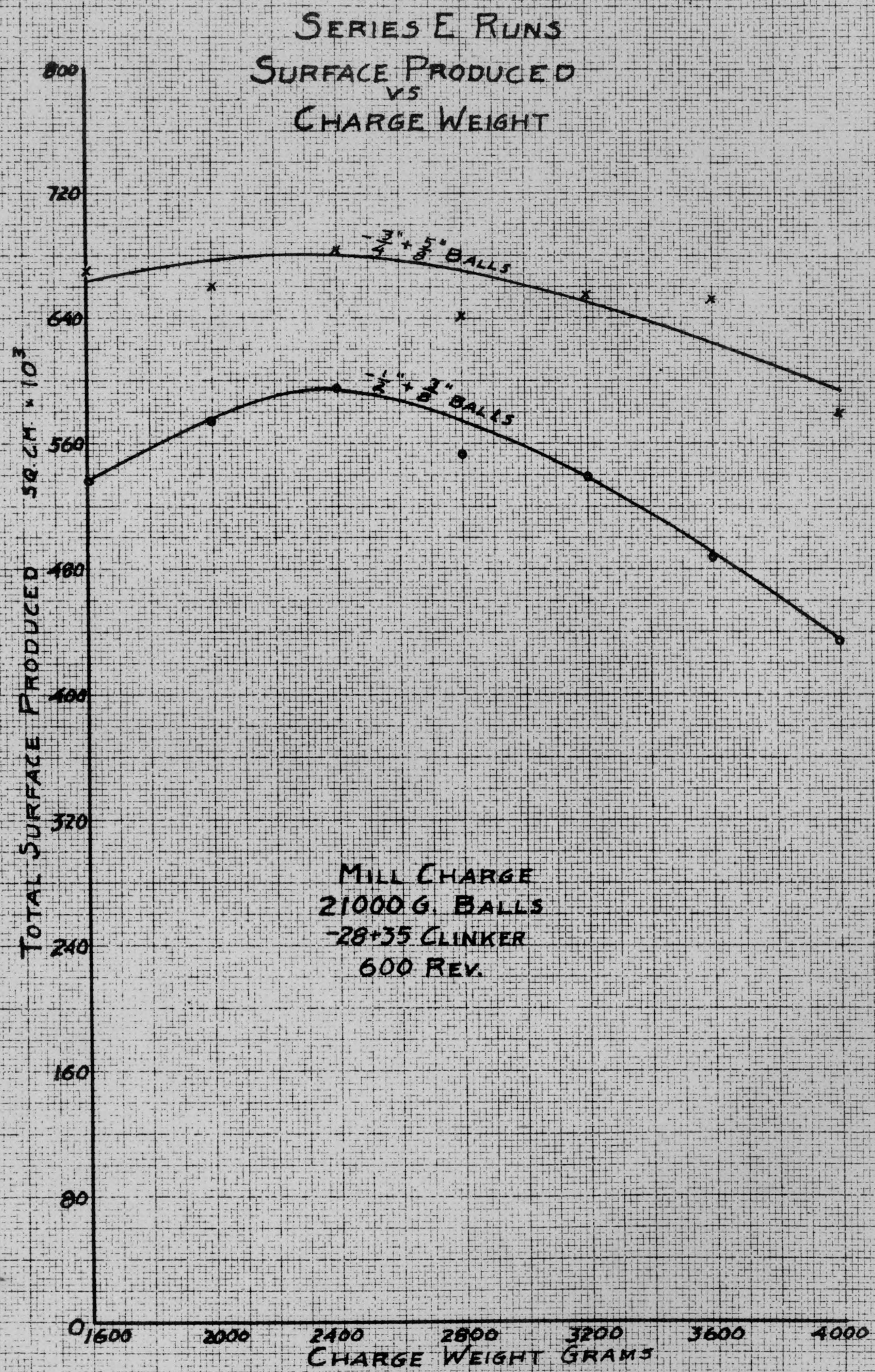

SERIES E RUNS
SURFACE PRODUCED
VS
CHARGE WEIGHT
$-\frac{3}{4}" + \frac{5}{8}"$ BALLS
$-\frac{1}{2}" + \frac{3}{8}"$ BALLS
MILL CHARGE
21000 G. BALLS
-28+35 CLINKER
600 REV.
TOTAL SURFACE PRODUCED SQ.CM. × 10³
CHARGE WEIGHT GRAMS
800
720
640
560
480
400
320
240
160
80
0
1600
2000
2400
2800
3200
3600
4000

FIG 43

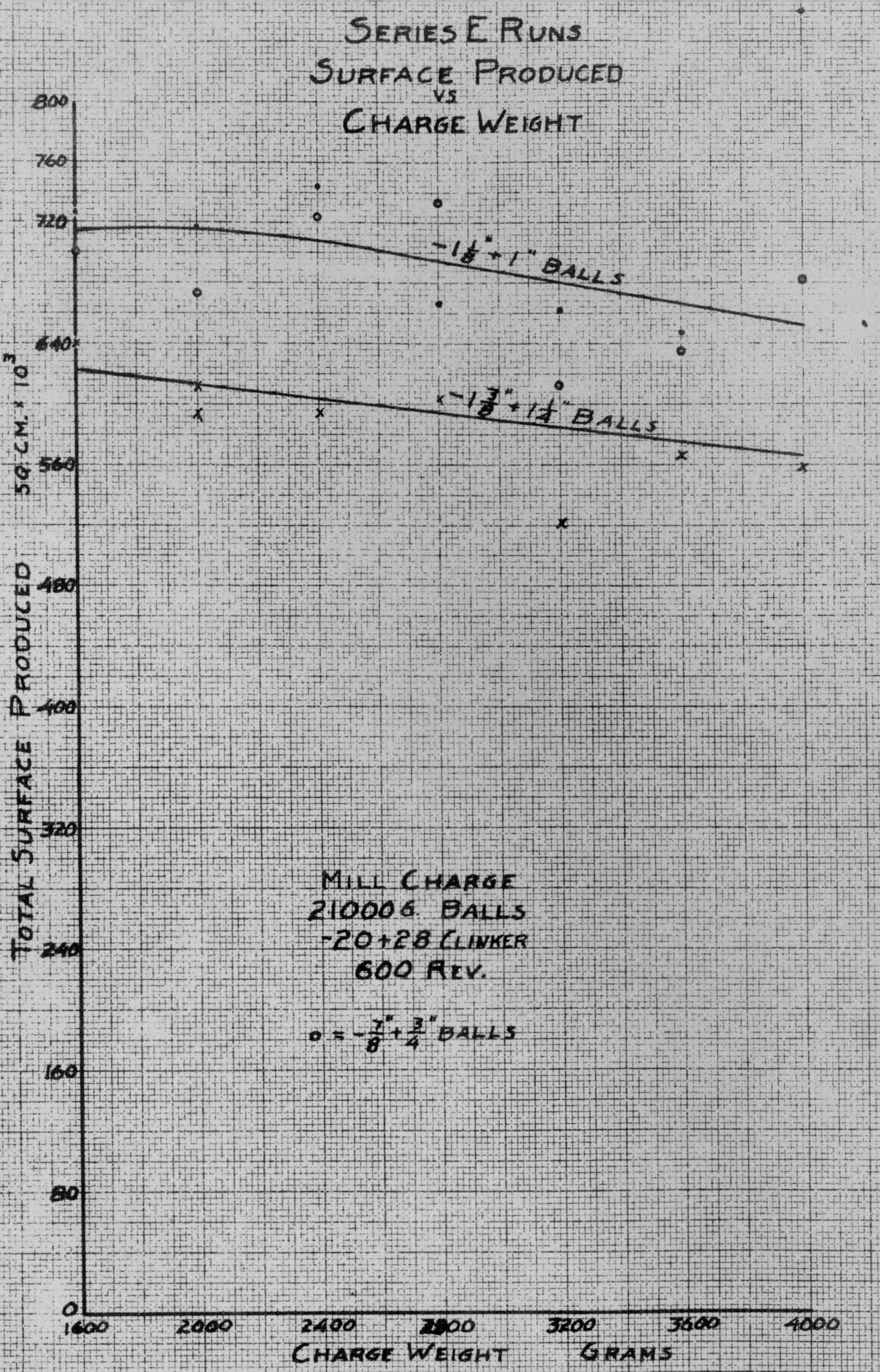

SERIES E RUNS
SURFACE PRODUCED
VS
CHARGE WEIGHT
TOTAL SURFACE PRODUCED SQ.CM. × 10³
800
760
720
640
560
480
400
320
240
160
80
0
1600
2000
2400
2800
3200
3600
4000
CHARGE WEIGHT GRAMS
-1 1/8" + 1" BALLS
-1 3/8" + 1 1/4" BALLS
MILL CHARGE
21000 G. BALLS
-20+28 CLINKER
600 REV.
o = -7/8" + 3/4" BALLS

FIG. 44

70. Discussion of Data. These curves show quite clearly that the grinding efficiency is not greatly affected by changing the amount of charge present. The data from the two smallest ball sizes (Fig. 43) is somewhat more consistent than the others. All groups indicate a tendency for the efficiency to drop with increasing charge weight. The smaller balls show optimum efficiency for charge weights whose volumes are about equal to the volume of the voids between the balls. The decrease in surface produced when the charge weight is high may be due to a change in the center of gravity of the combined mass of balls and clinker. If more material is in the mill than is required to fill the voids between the balls, and if it is distributed evenly through the balls, the center of gravity of the mill contents will rise. If the moment of the mass about the center line of the mill is smaller than it was when there was just enough charge to fill the voids, then the power input would be expected to be smaller in the former case. If this is true, a decrease in surface produced would result, even if the efficiency remained constant, because the energy input would be lower. For example, calculations based on the actual and apparent densities of balls and clinker show that it would require 2790 grams of -28 + 35 clinker to fill the voids in the 21,000 gram load of -3/4" + 5/8" balls. With this load the center of gravity of the total charge would be 3.17" from the center line of the mill, and the moment would be 166 inch-pounds. If the clinker charge wasincreased to 4000 grams, the center of gravity would be 2.72" from the center. The moment would be 149 inch-pounds, or a decrease of 10.3 %. Values taken from the curve corresponding to these conditions Fig. (43) show a decrease of 11.2 % in the surface produced with

a 4000 gram charge over that with a 2800 gram charge. Consequently, in this case, the efficiency of grinding remained constant although the surface produced decreased as the clinker charge increased. The slopes of the curves shown in Figs. (43) and (44) decrease as ball size increases, i.e., the curve for the -½"+3/8 " balls is the steepest, while that for the -1 3/8"+ 1¼" balls is the most nearly horizontal. This is again probably due to the change in the center of gravity of the mass. The apparent density of the balls also decreases as the size increases. The value for the -½"+3/8" balls is 5.0 and that for the -1 3/8" + 1¼", 4.6 This means that the volume of the voids is smaller for the small balls, hence less clinker is required to fill it and, with a given maximum charge, more is left over to increase the total volume of material in the mill and thus raise the center of gravity. For this reason the percent decrease in power required would be largest with the smallest balls and smallest with the largest ones. Likewise, the decrease in surface produced would be the largest with the smallest balls and vice versa.

71. Conclusions: The conclusions to be drawn from the above data and discussions are:

(1) The total surface produced by a ball mill remains practically constant for charge weights between 50 % and 100 % of the amount required to fill the voids between the balls.

(2) The total surface produced decreases a small amount with increase in charge weight above that required to fill the voids. This decrease is accompanied by a corresponding decrease in power required by the mill.

(3) The grinding efficiency is not affected by such variations.

72. Effect of Change in Charge Weight Upon Size Distribution of Product. The above conclusions suggest a corollary. If the total surface produced is practically constant for different charge weights, then the smaller charges must be ground finer to yield the same surface. If this is true the percentage of -200 mesh material in the product will be higher for the smaller charges. This is shown to be the case by Figs. (45) to (47). These are Cumulative Distribution Curves for the complete size range, plotted with the ordinate representing the percent of the weight smaller than the given size. The material containing the highest amount of small material is consequently represented by the highest curve.

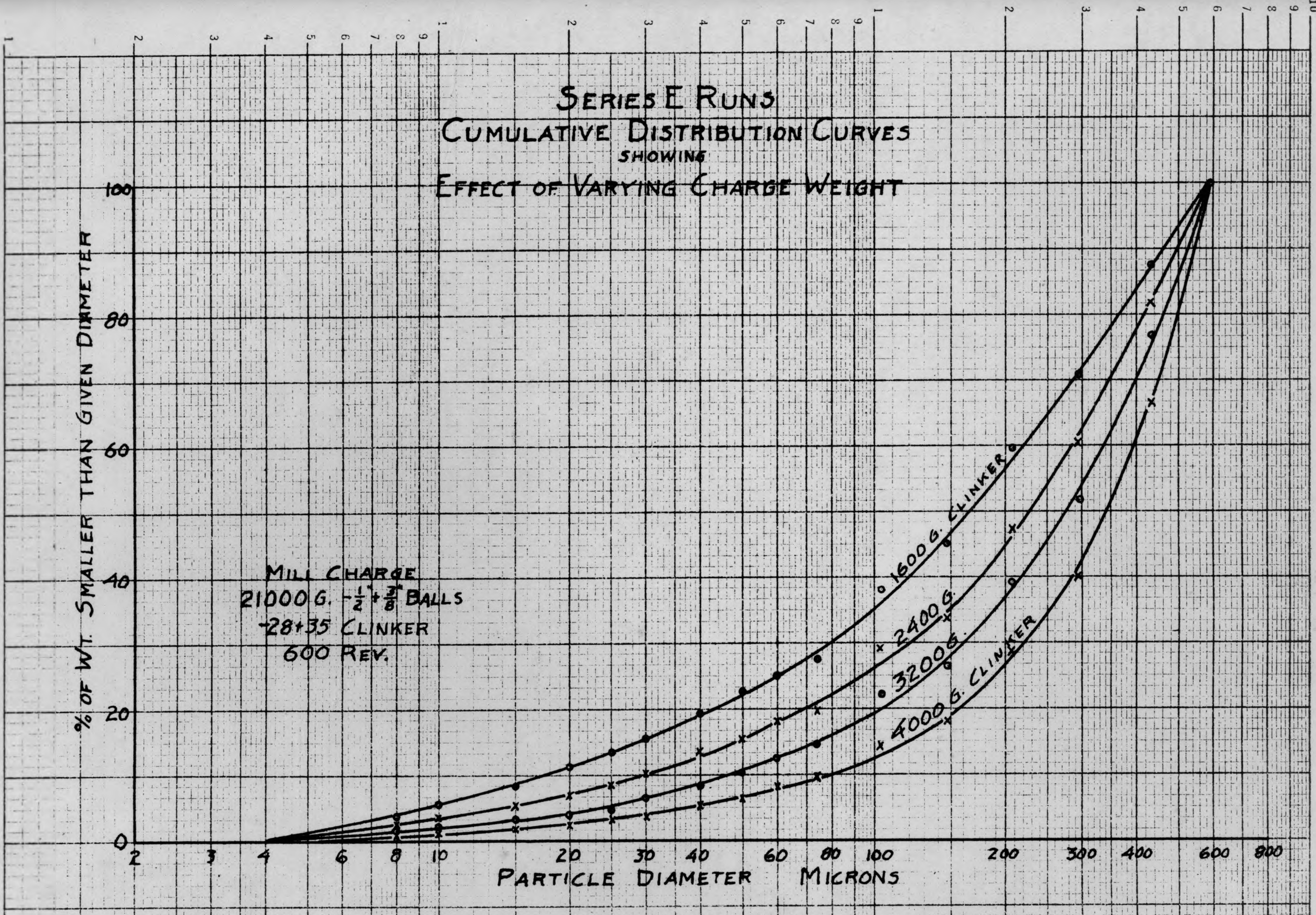
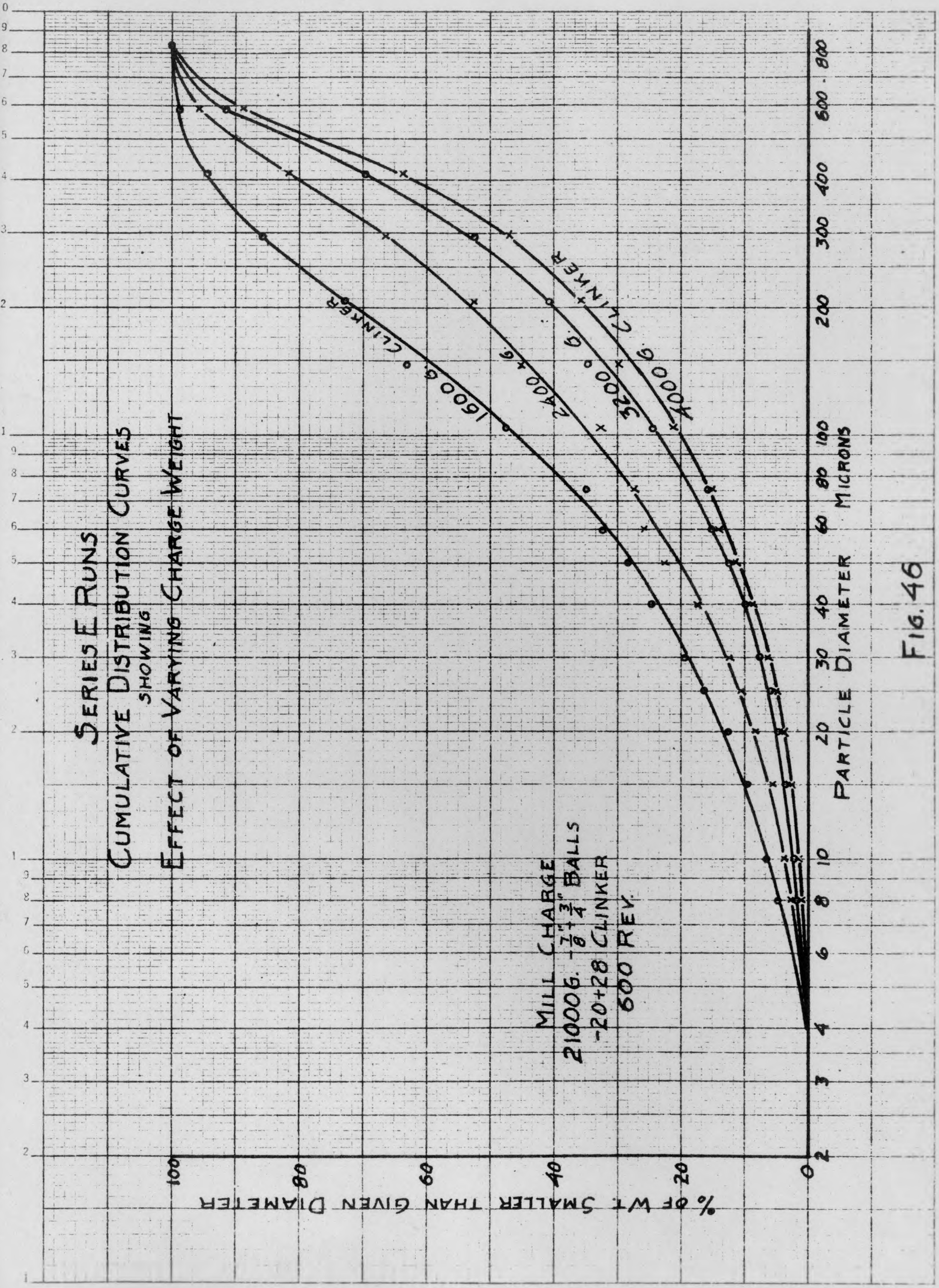

SERIES E RUNS
CUMULATIVE DISTRIBUTION CURVES
SHOWING
EFFECT OF VARYING CHARGE WEIGHT
MILL CHARGE
21000 G. -$\frac{1}{2}$"+$\frac{3}{8}$" BALLS
-28+35 CLINKER
600 REV.
1600 G. CLINKER
2400 G.
3200 G.
4000 G. CLINKER
% OF WT. SMALLER THAN GIVEN DIAMETER
0
20
40
60
80
100
PARTICLE DIAMETER MICRONS
2
3
4
6
8
10
20
30
40
60
80
100
200
300
400
600
800

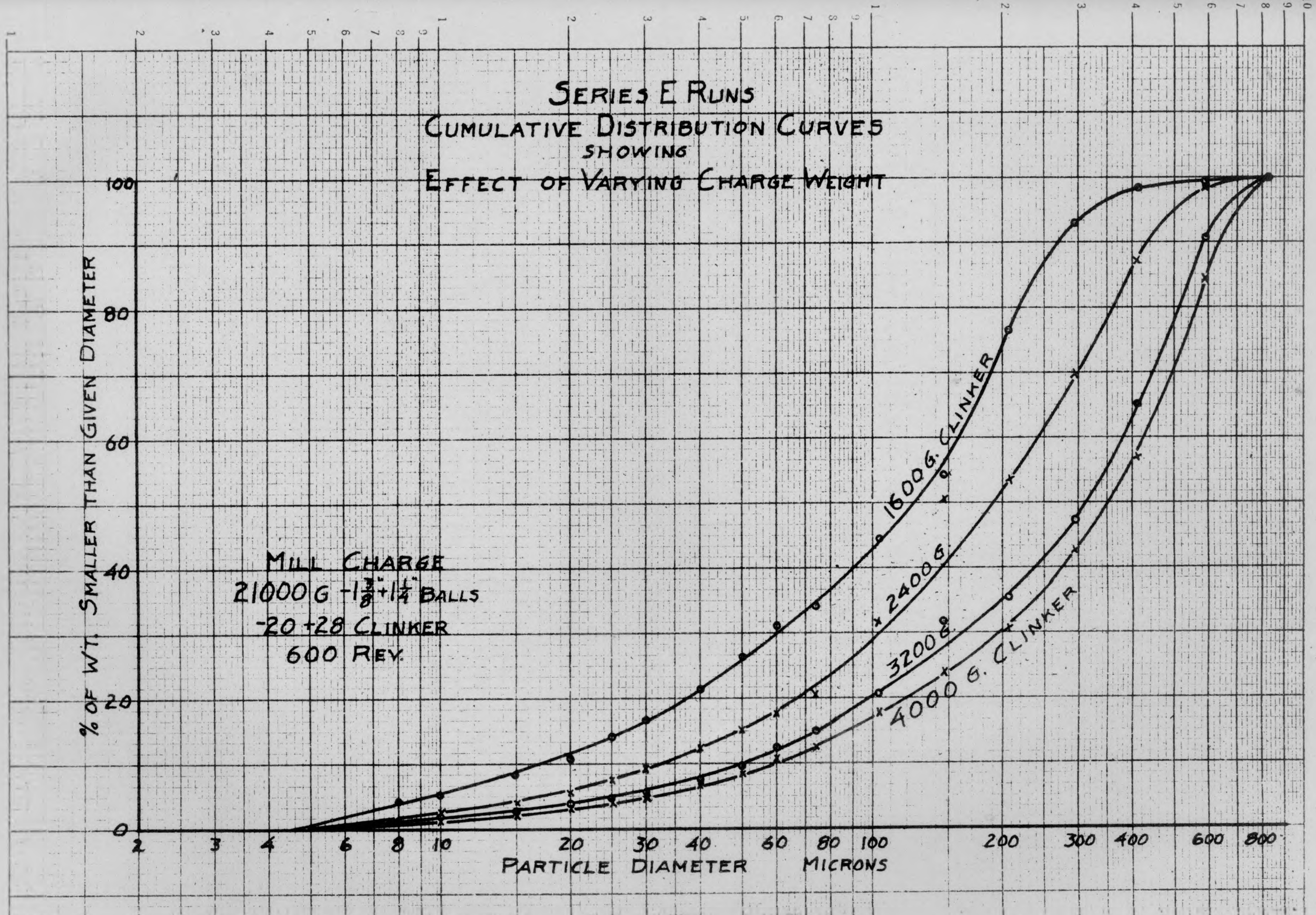
FIG. 45



SERIES E RUNS
CUMULATIVE DISTRIBUTION CURVES
SHOWING
EFFECT OF VARYING CHARGE WEIGHT

MILL CHARGE
21000 G. $-\frac{7}{8}"+\frac{3}{4}"$ BALLS
-20+28 CLINKER
600 REV.

FIG. 46



SERIES E RUNS
CUMULATIVE DISTRIBUTION CURVES
SHOWING
EFFECT OF VARYING CHARGE WEIGHT

MILL CHARGE
21000 G - 1$\frac{3}{8}$" + 1$\frac{1}{4}$" BALLS
-20 +28 CLINKER
600 REV.

FIG. 47

III. Effect of the Ratio of Particle Diameter to Ball Diameter Upon the Size Distribution of the Product.

73. General Discussion. Another statement (19) often encountered in treatises on ball and tube mills refers to methods of regulating the size distribution of the product. It is said that if a product of uniform size is desired, a large ball should be used and relatively large sized material fed to the mill. Conversely, if it is desired to have a high amount of "flour" in the product, small balls should be used with correspondingly small sized feed particles. Both Series E and No.2 Time Series offer data which may be used in testing this statement.

74. Data FromSeries E. Series E will be presented first, since the conditions of operation were simpler because one variable, the length of time of grinding, was held constant. The data is presented in the form of percent per micron charts because they show the size distribution of the product most clearly. The point of interest here is not the size distribution over the entire range, but only that of the material which will pass the 200 mesh sieve. Therefore the charts are plotted from the Microneter data only, i.e., they are on the basis of 100 % minus 200 mesh. To prevent confusion from too much data, four ball sizes were selected, and the results of runs made with these sizes on three representative clinker sizes were compared. Figs. (48), (49) and (50) are the charts obtained. The ball and clinker sizes not represented yield results that fit in, in proper order, between the sizes shown.

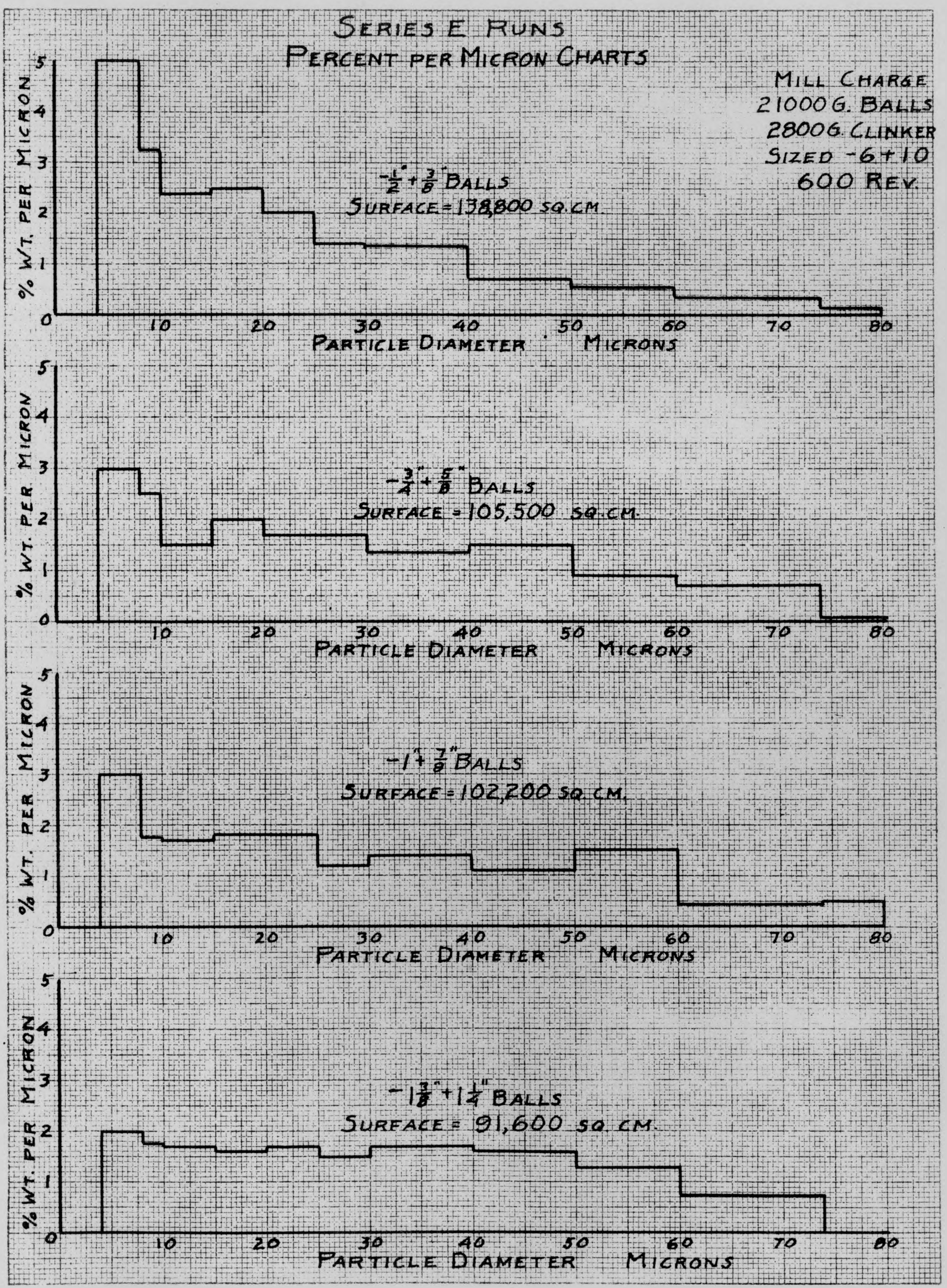

SERIES E RUNS
PERCENT PER MICRON CHARTS
MILL CHARGE
21000 G. BALLS
2800 G. CLINKER
SIZED -6+10
600 REV.
-1/2" + 3/8" BALLS
SURFACE = 138,800 SQ. CM.
-3/4" + 5/8" BALLS
SURFACE = 105,500 SQ. CM.
-1" + 7/8" BALLS
SURFACE = 102,200 SQ. CM.
-1 3/8" + 1 1/4" BALLS
SURFACE = 91,600 SQ. CM.
% WT. PER MICRON
0
1
2
3
4
5
PARTICLE DIAMETER MICRONS
10
20
30
40
50
60
70
80

FIG. 48

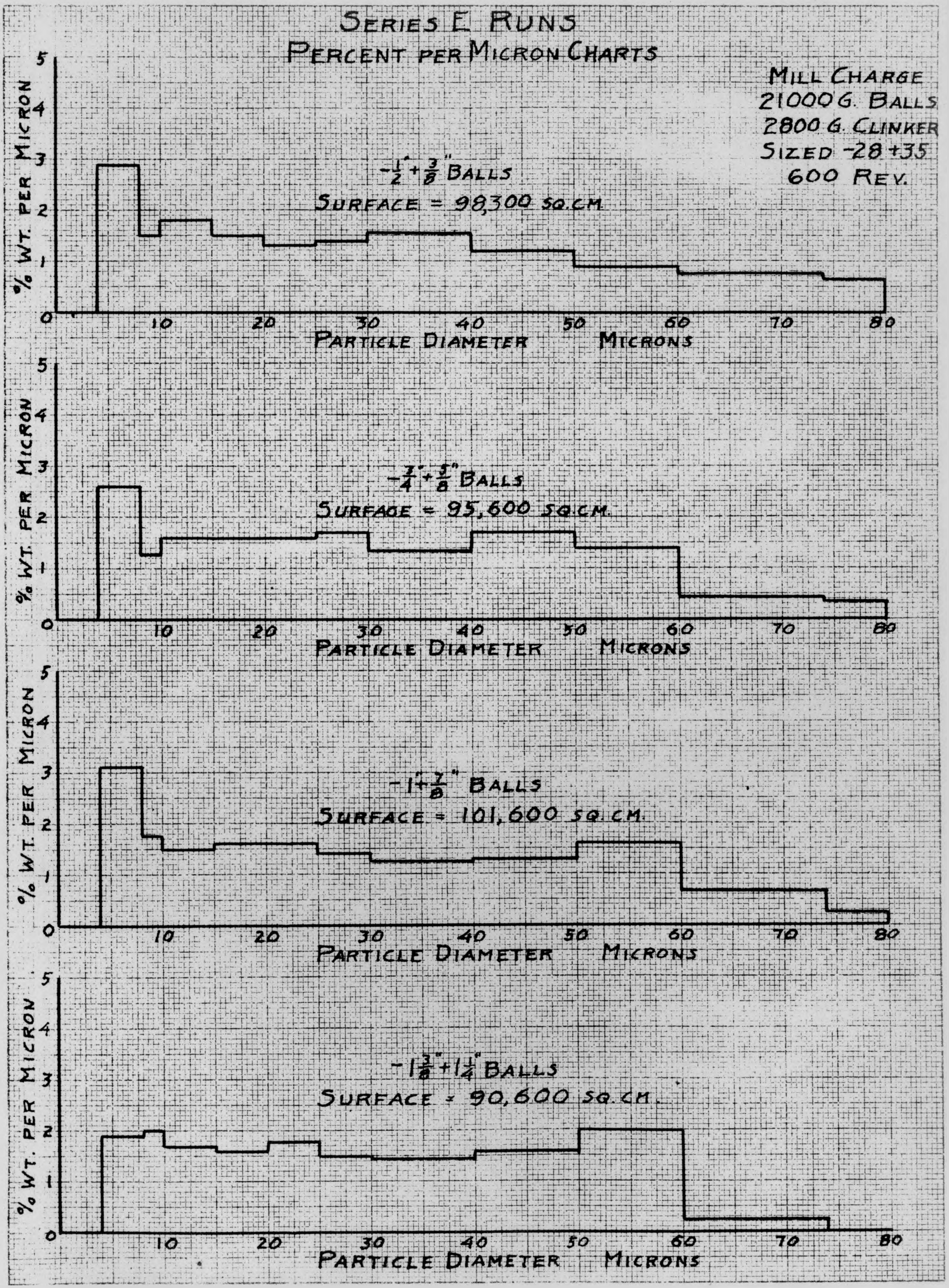

SERIES E RUNS
PERCENT PER MICRON CHARTS
MILL CHARGE
21000 G. BALLS
2800 G. CLINKER
SIZED -28 +35
600 REV.
$-\frac{1}{2}" + \frac{3}{8}"$ BALLS
SURFACE = 98,300 SQ.CM.
$-\frac{3}{4}" + \frac{5}{8}"$ BALLS
SURFACE = 95,600 SQ.CM.
$-1" + \frac{7}{8}"$ BALLS
SURFACE = 101,600 SQ.CM.
$-1\frac{3}{8}" + 1\frac{1}{4}"$ BALLS
SURFACE = 90,600 SQ.CM.
% WT. PER MICRON
0
1
2
3
4
5
PARTICLE DIAMETER MICRONS
10
20
30
40
50
60
70
80

FIG. 49

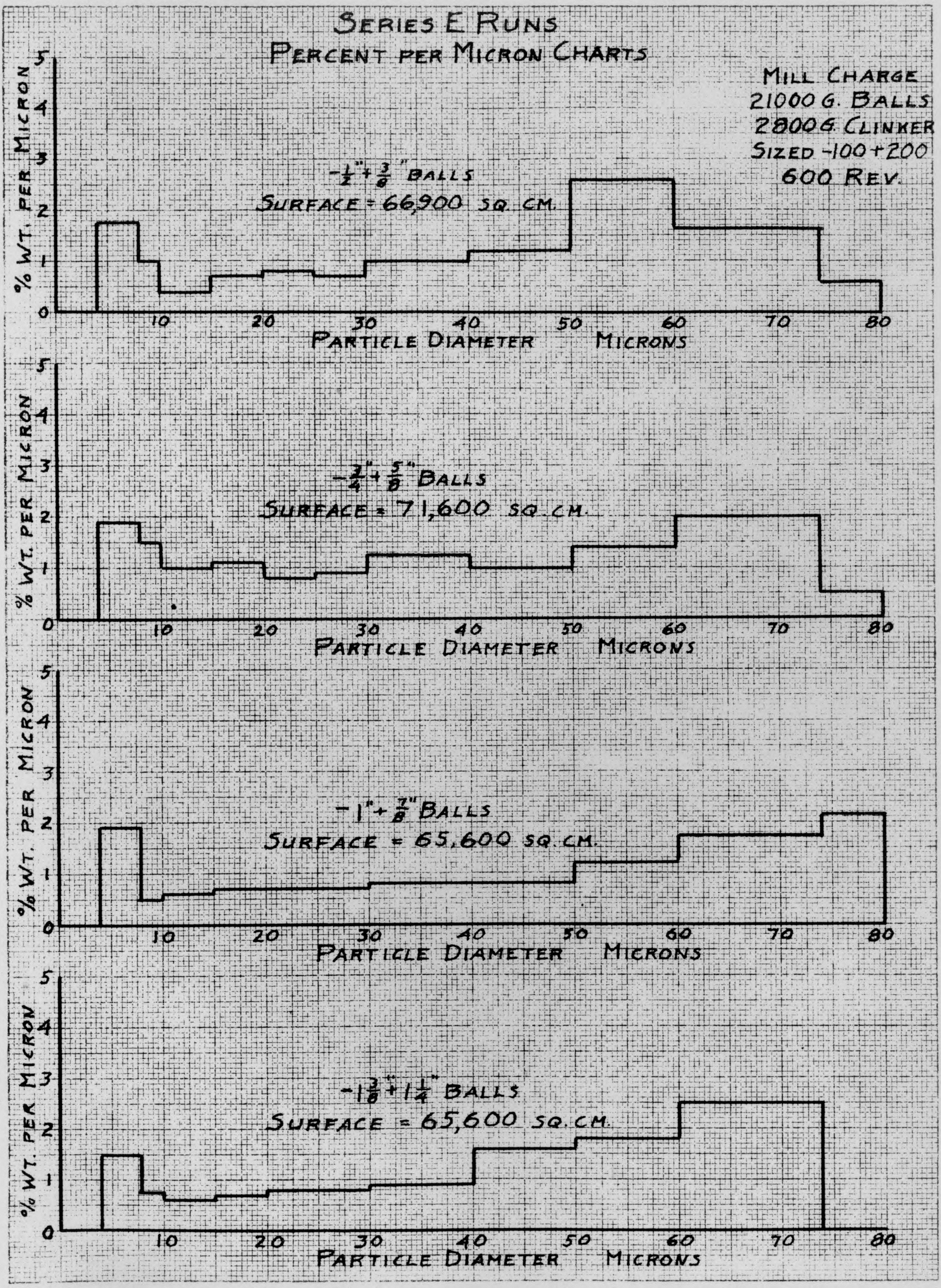

SERIES E RUNS
PERCENT PER MICRON CHARTS
MILL CHARGE
21000 G. BALLS
2800 G. CLINKER
SIZED -100+200
600 REV.
$-\frac{1}{2}" + \frac{3}{8}"$ BALLS
SURFACE = 66,900 SQ. CM.
$-\frac{3}{4}" + \frac{5}{8}"$ BALLS
SURFACE = 71,600 SQ. CM.
$-1" + \frac{7}{8}"$ BALLS
SURFACE = 65,600 SQ. CM.
$-1\frac{3}{8}" + 1\frac{1}{4}"$ BALLS
SURFACE = 65,600 SQ. CM.
% WT. PER MICRON
0
1
2
3
4
5
10
20
30
40
50
60
70
80
PARTICLE DIAMETER MICRONS

FIG. 50

75. Discussion of Data from Series E. Fig. (48) shows the action of the various sizes of balls of coarse material. The differences are very marked. The smallest balls produce a large amount of very fine material but very little between 40 and 80 microns, while the largest ones yield a product which has a uniform size gradation over the whole range. The intermediate ball sizes give material of intermediate size gradation. Fig.(49) illustrates the action of the balls on a smaller material. The size gradations still show differences in the same direction but the magnitudes of the differences are much less. The size distribution of the material ground with the large balls is similar to that of the larger material run with the same balls. The amount of very fine material produced is much less with the charge of smaller initial particle size. Fig. (50) presents the data from the runs on the finest material. It is seen that there is now little difference between any of the runs, irrespective of ball size. The values of the surfaces are shown on each diagram. They corroborate the statements just made. It would be expected that the conditions yielding the largest amount of very fine material would produce the smallest total amount finer than 200 mesh, because there is so much more surface represented by a given weight of the -200 material. This is the actual state of affairs and it is accentuated somewhat by the lower grinding efficiency of the small balls when used with the large particles. The curves shown in Figs.(37) to (44) indicate this decrease in efficiency.

76. Data From No.2 Time Runs. No. 2 Time Runs offer the possibility of obtaining information about the effect of the length of time of grinding upon the size distribution of the product

from charges of different initial particle sizes. Three different ball sizes run with two different clinker sizes for three varying lengths of time have been chosen as representative of the performance of these series. The data is represented in Figs. (51) to (56). It is in the same form as that from Series E, which has just been discussed (Par.74,75. Figs. 48 to 50). The statement made about Series E is also true for this group of runs,i.e., that the data shown is truly representative of the entire series.

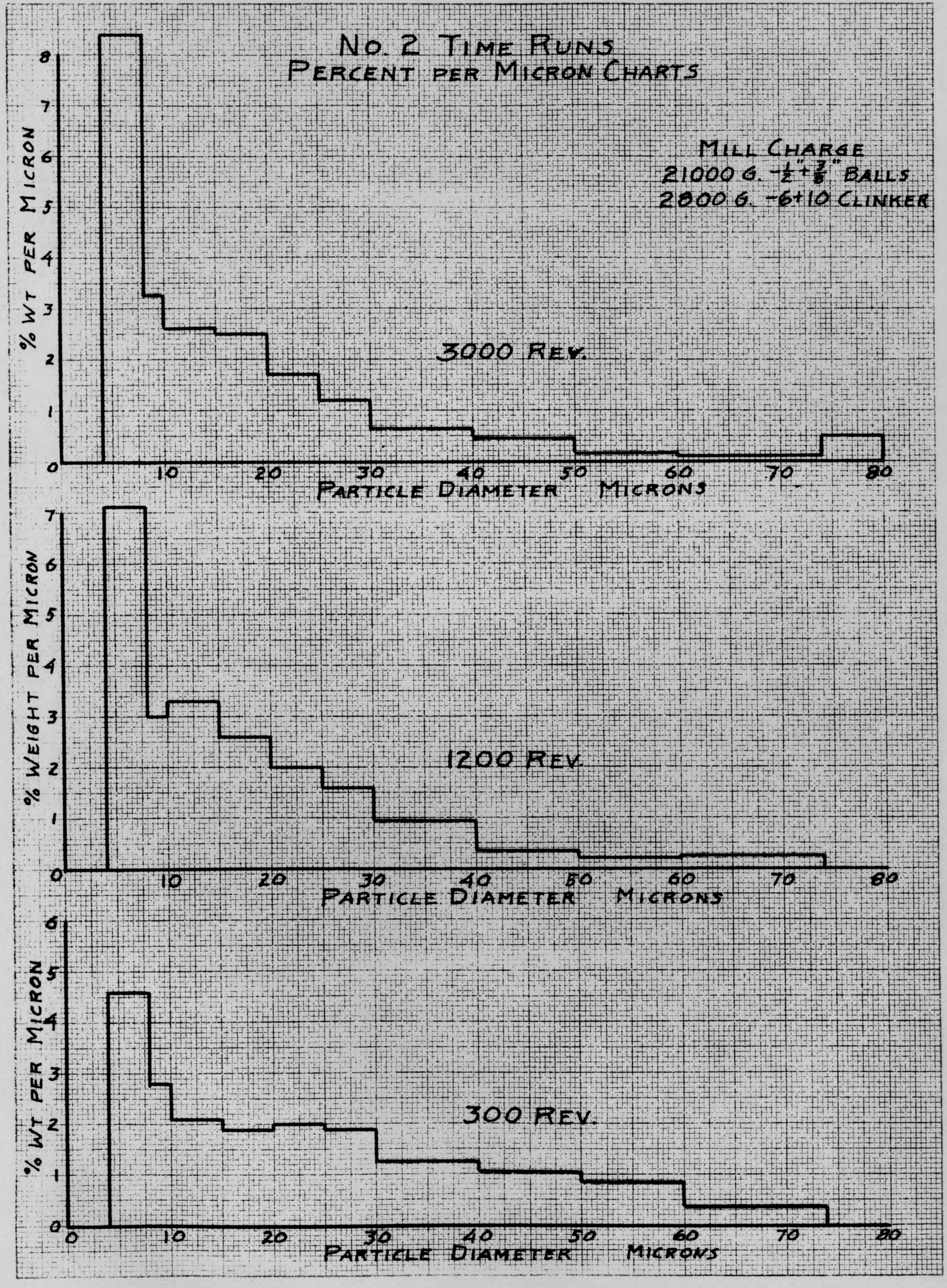

No. 2 Time Runs
Percent per Micron Charts
Mill Charge
21000 G. $-\frac{1}{2}"+\frac{3}{8}"$ Balls
2800 G. -6+10 Clinker
3000 Rev.
% Wt per Micron
0
1
2
3
4
5
6
7
8
10
20
30
40
50
60
70
80
Particle Diameter Microns
1200 Rev.
% Weight per Micron
0
1
2
3
4
5
6
7
10
20
30
40
50
60
70
80
Particle Diameter Microns
300 Rev.
% Wt per Micron
0
1
2
3
4
5
6
0
10
20
30
40
50
60
70
80
Particle Diameter Microns

FIG. 51

No. 2 Time Runs
Percent per Micron Charts

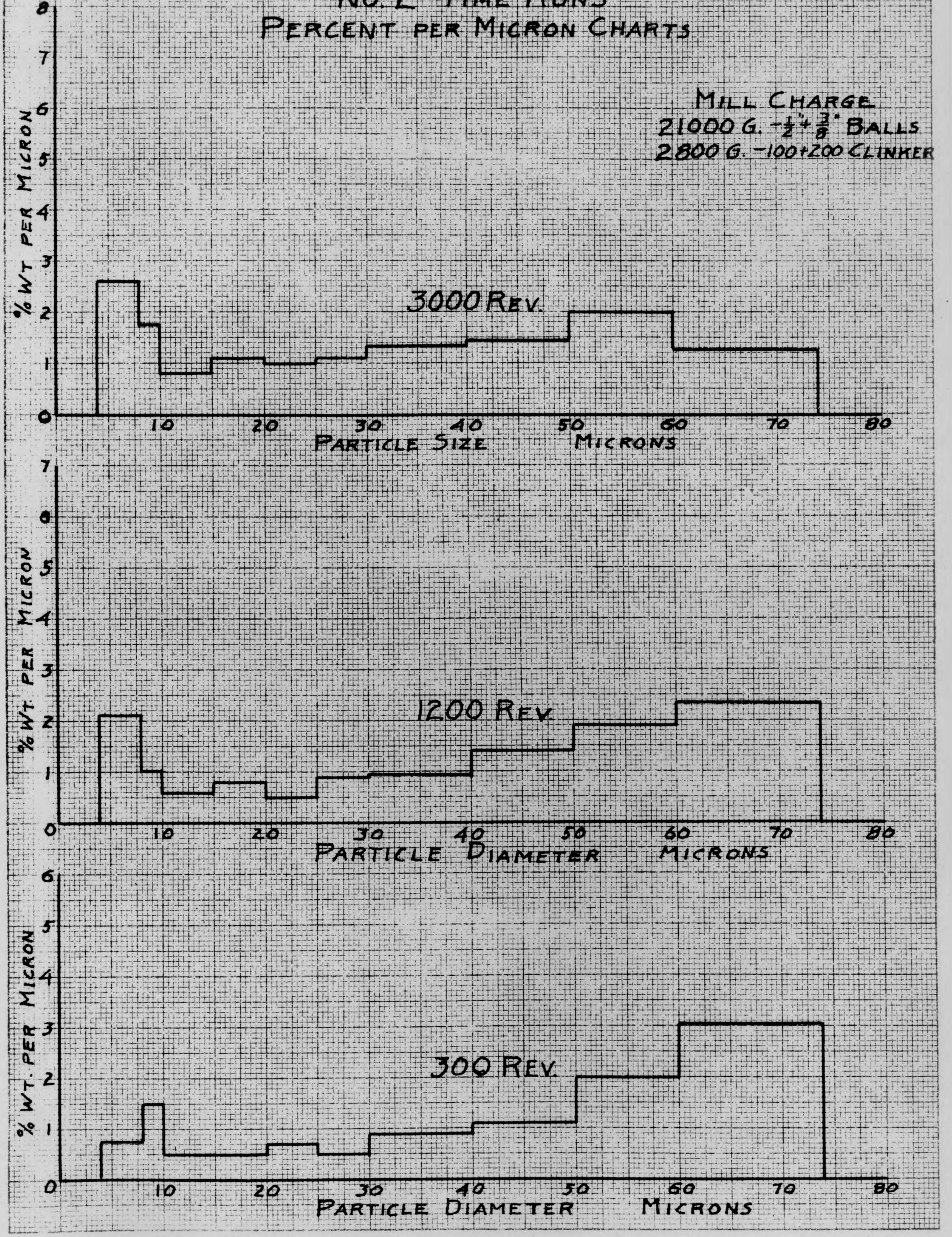
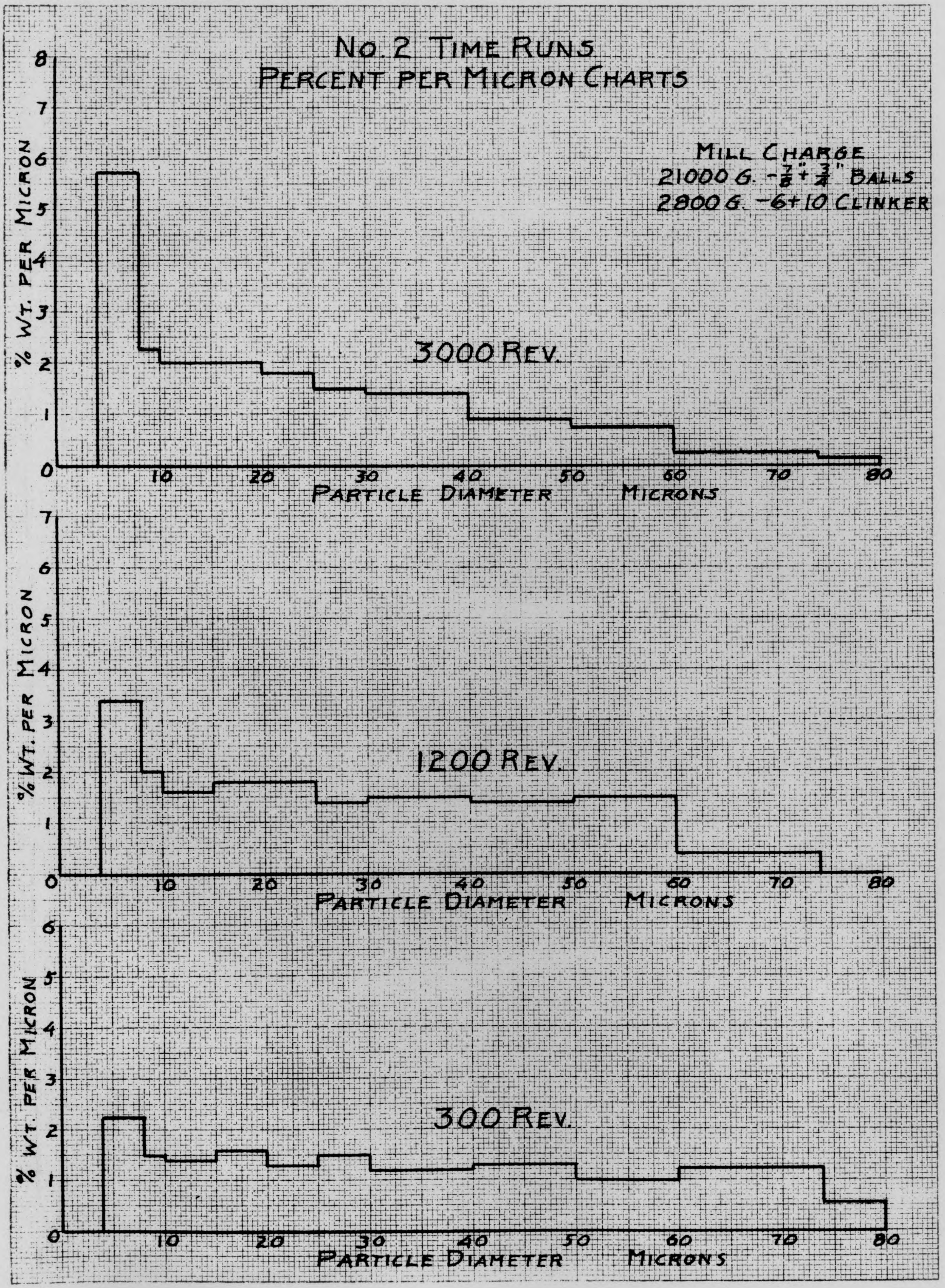


Fig. 52



NO. 2 TIME RUNS
PERCENT PER MICRON CHARTS

MILL CHARGE
21000 G. $-\frac{7}{8}"+\frac{3}{4}"$ BALLS
2800 G. −6+10 CLINKER

FIG. 53

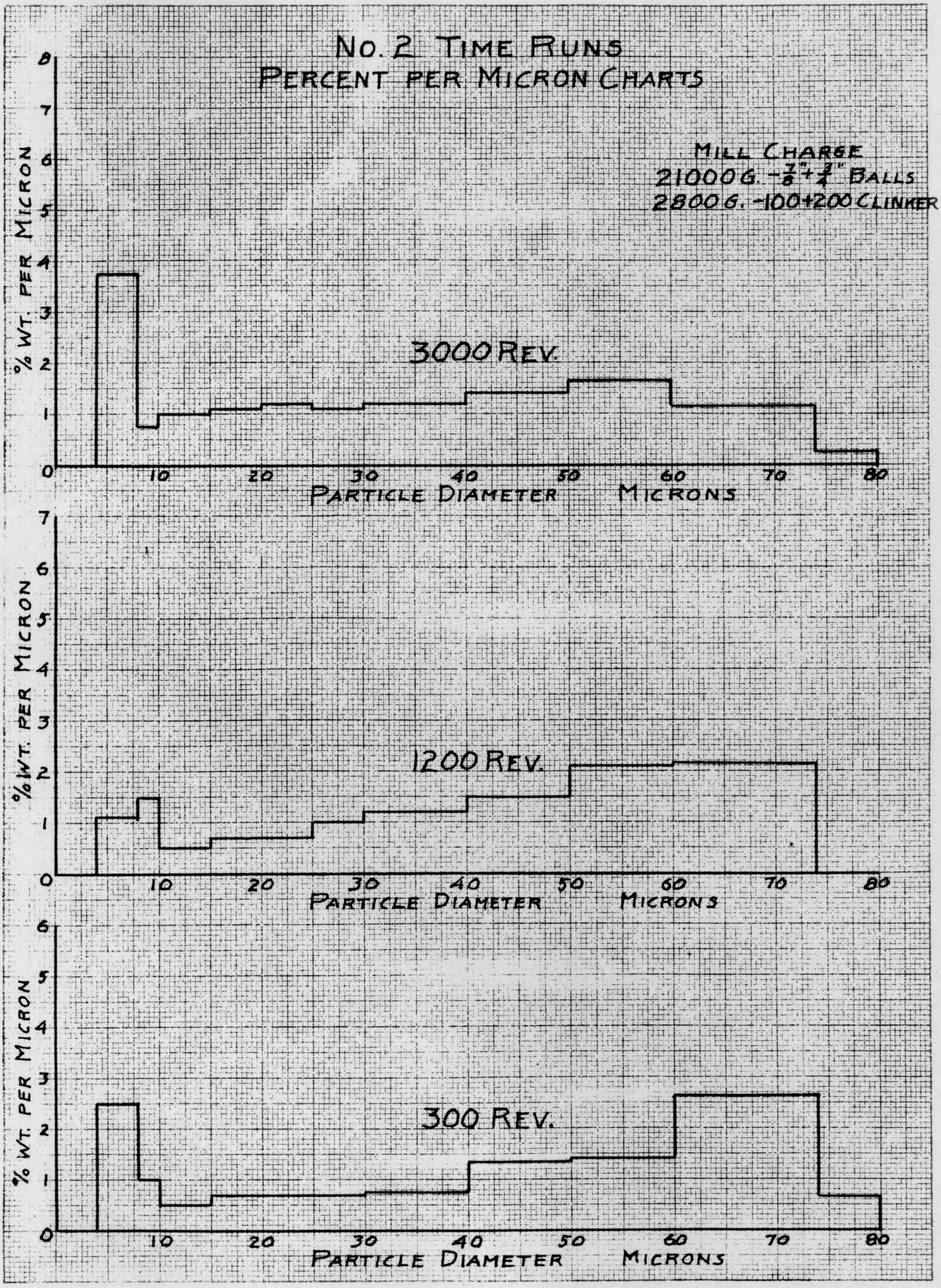

No. 2 Time Runs
Percent per Micron Charts
Mill Charge
21000 G. -7/8" + 3/4" Balls
2800 G. -100+200 Clinker
3000 Rev.
1200 Rev.
300 Rev.
% Wt. per Micron
0
1
2
3
4
5
6
7
8
10
20
30
40
50
60
70
80
Particle Diameter Microns

FIG. 54

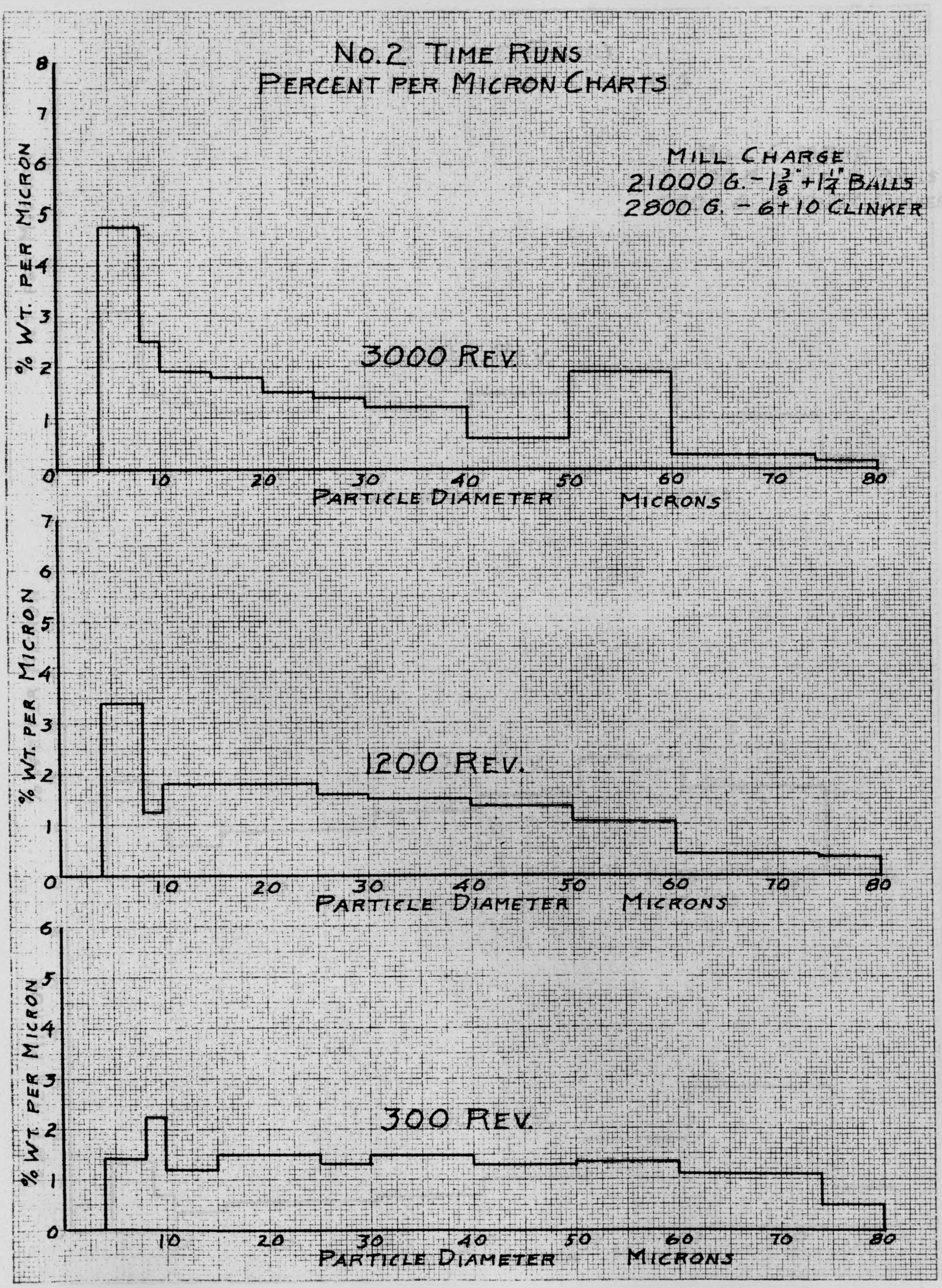

No.2 Time Runs
Percent per Micron Charts
Mill Charge
21000 G. $-1\frac{3}{8}$" $+1\frac{1}{4}$" Balls
2800 G. $-6+10$ Clinker
3000 Rev.
1200 Rev.
300 Rev.
% Wt. per Micron
0
1
2
3
4
5
6
7
8
10
20
30
40
50
60
70
80
Particle Diameter
Microns

FIG. 55

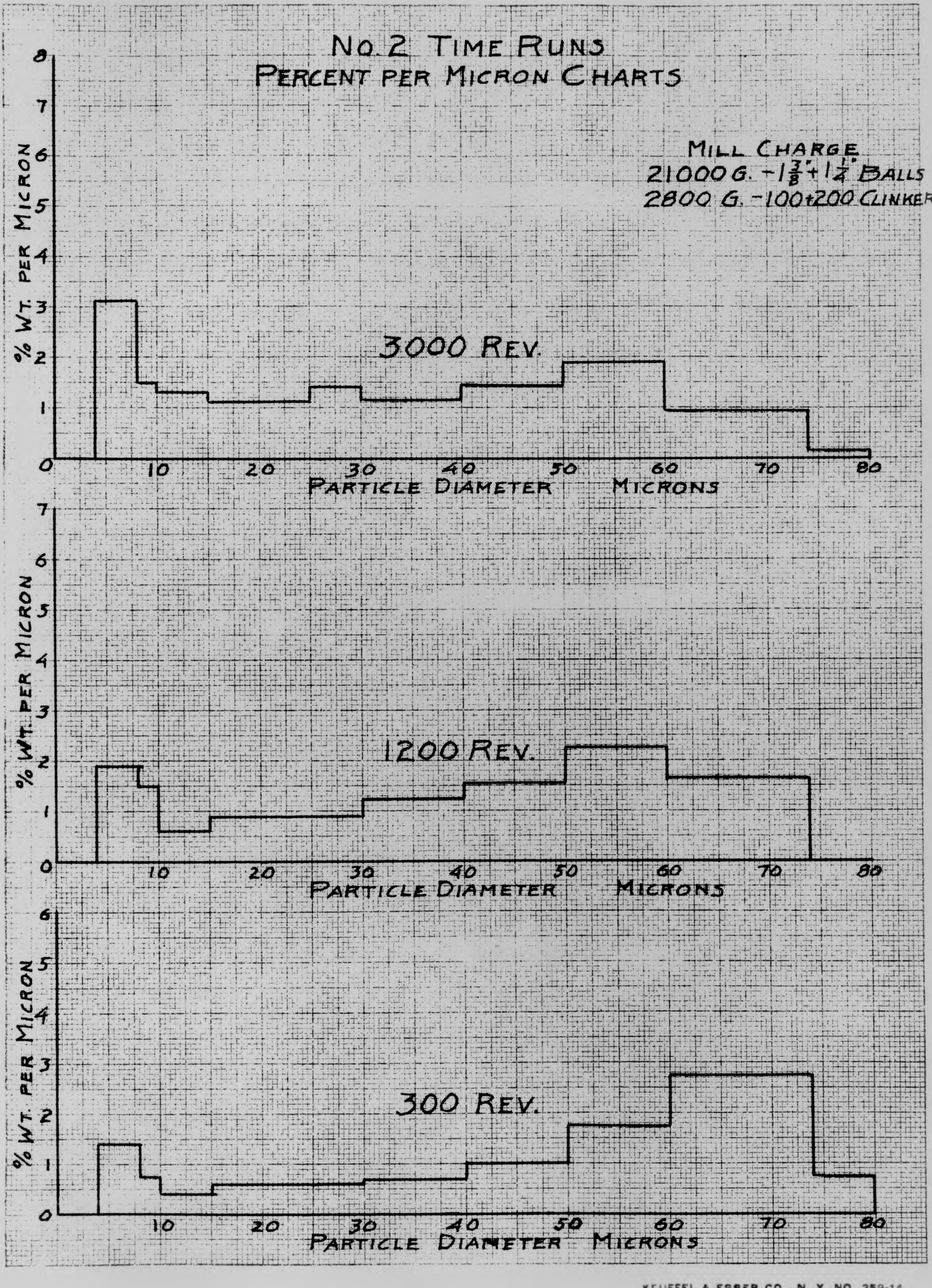

No. 2 Time Runs
Percent per Micron Charts
Mill Charge
21000 G. -1 3/8" + 1 1/4" Balls
2800 G. -100+200 Clinker
% Wt. per Micron
0
1
2
3
4
5
6
7
8
3000 Rev.
10
20
30
40
50
60
70
80
Particle Diameter Microns
% Wt. per Micron
0
1
2
3
4
5
6
7
1200 Rev.
10
20
30
40
50
60
70
80
Particle Diameter Microns
% Wt. per Micron
0
1
2
3
4
5
6
300 Rev.
10
20
30
40
50
60
70
80
Particle Diameter Microns

FIG. 56

77. Discussion of Data. Figs.5I and 52 show the results obtained when the grinding is done with small balls. They demonstrate that the effect of increasing the time of grinding is to accentuate the conditions observed at the end of the first short period. The relative shapes of the size distribution charts remain the same for all the various lengths of runs, and indeed, are the same as those obtained from Series E runs (Fig.48-50). The charts for the runs with the larger balls (Fig.53,54 and 55,56)., also correspond closely to those for similar ball sizes in Series E. They likewise show the tendency seen in Figs.5I and 52 to maintain the same general size distribution irrespective of time of grinding, although on the average the amount of -I0 micron material increases with increasing time. This effect is more noticeable with with the larger balls acting on the fine material, than with the small balls under the same conditions. Consequently for the longer runs the large balls produce the highest amount of very fine material when the intial particle size is small.

Since the size gradation of the -200 mesh fraction of the product did not change very much, it follows that the energy represented by the increase in the time of grinding must have been expended in producing a larger amount of this material. This was seen to be the case in Figs. (39),(40),(41), which were plotted with the data obtained from this series. Practically all of the surface of the material is in the fraction smaller than 200 mesh. The total surface is therefore approximated by the product of the surface of the-200 mesh material by its percentage weight. Since the total surface increased linearly with the time of grinding, the amount of -200 mesh material likewise must have increased.

78. Conclusions. These two sets of runs show that,

(1) The highest percentage of "flour" is produced when material of large particle size is ground with small balls.

(2) Very little flour is produced when small balls are used to grind material which is initially quite fine. Large balls produce slightly more flour under similar conditions, especially with long grinding periods.

(3) The time of grinding has comparatively little effect upon the size gradation of the part of the product smaller than 200 mesh. Increased time of grinding serves primarily to produce more of the same sized material.

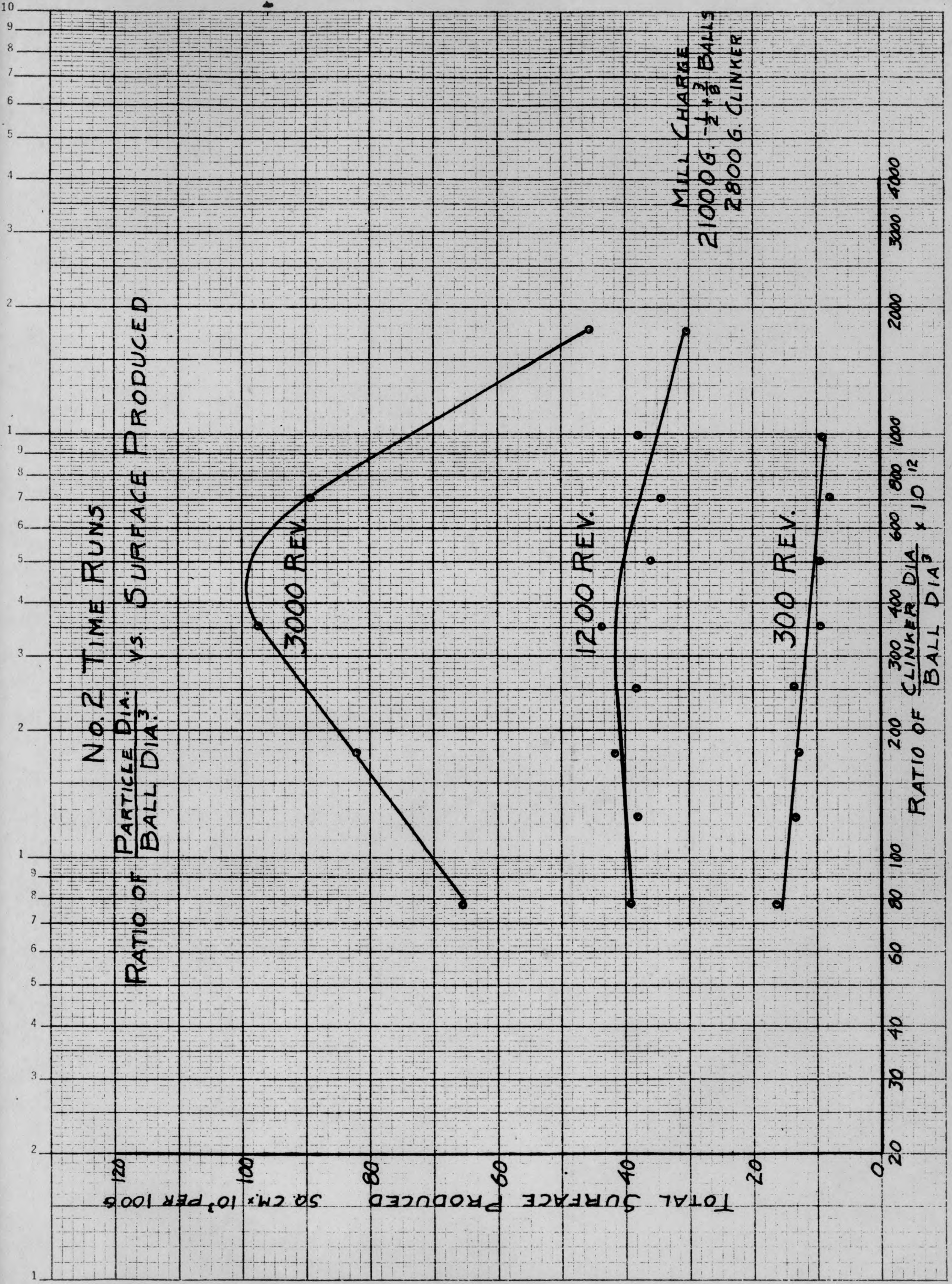
These conclusions are practically the reverse of the generally accepted ideas on the subject.

IV. Effect of Varying The Ratio of $\frac{\text{Particle Diameter}}{\text{Ball Diameter}}$ on the Efficiency of Grinding.

79. General Discussion. All of the preceding results have shown that the relation between the size of the particles being ground and the balls doing the grinding plays an important part in the results obtained. An attempt has been made to develop a general index for this effect which could be applied to the different sizes of balls and particles worked with. There are many kinds of relations existing between these two components of the mill charge. For example, it might be the relation between the two diameters, surfaces, or volumes. On the other hand, it

could be that between the diameter of one and the surface of the other, or any other similar combination. However, it appeared very likely that the weight of the balls would certainly be one of the factors, and probably that either the diameter or the surface of the particles would be the other. A little consideration of the data showed that fo these latter, the diameter was the most probable. Since the density of the steel in the balls is constant the weight is directly proportional to the volume. This in turn is proportional to the cube of the diameter. Consequently the ratio $\frac{\text{Particle diameter}}{\text{Ball diameter}^3}$ is directly proportional to $\frac{\text{Particle diameter}}{\text{Ball weight}}$.

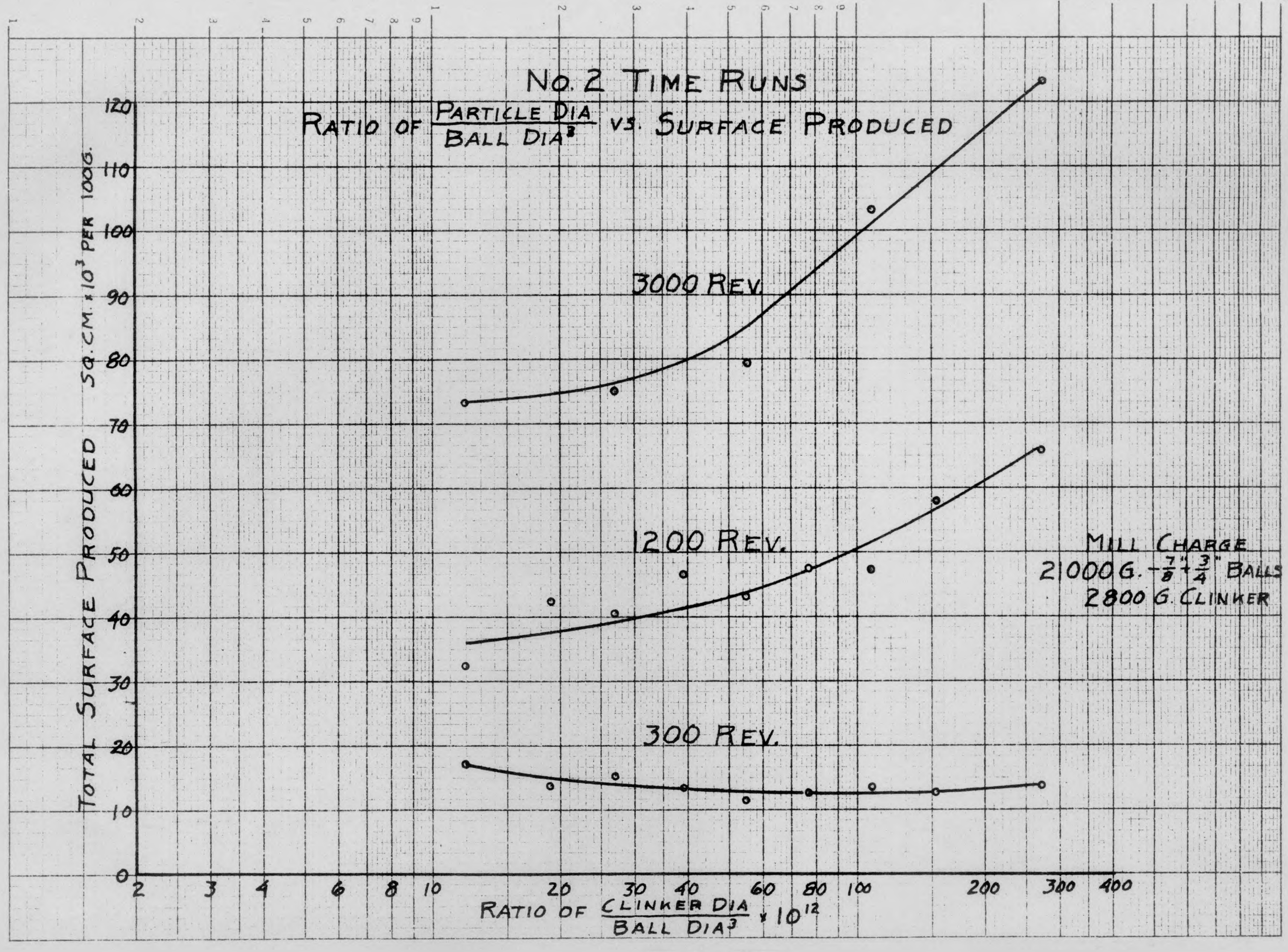
80. <u>Data from No.2 Time Runs.</u> No.2 Time Runs have again furnished the data used in investigating the influence of this ratio. This is shown in Figs. (57),(58) and (59). The coordinates chosen,(namely, the ratio of $\frac{\text{Particle diameter}}{\text{Ball diameter}^3}$ for abscissa and surface produced for ordinate) serve to demonstrate the effects of changes in this ratio upon the grinding efficiency. The same ball sizes are used as before. The curves represent different lengths of runs, and each point on a curve indicates a charge of different particle size.



No. 2 Time Runs

Ratio of $\frac{\text{Particle Dia.}}{\text{Ball Dia.}^3}$ vs. Surface Produced

FIG. 57



No. 2 Time Runs

Ratio of $\frac{\text{Particle Dia}}{\text{Ball Dia}^3}$ vs. Surface Produced

FIG. 58

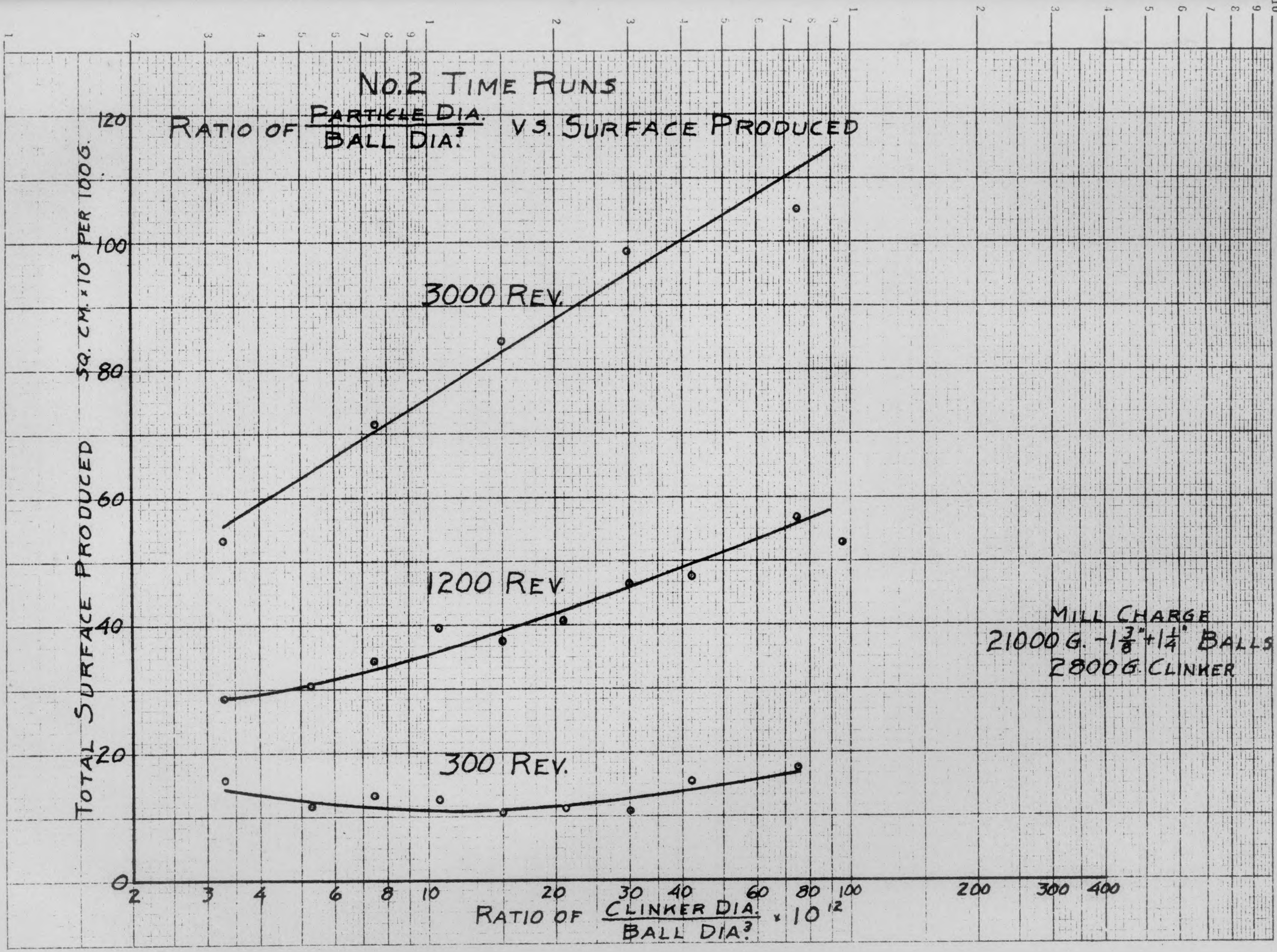

No.2 TIME RUNS
RATIO OF PARTICLE DIA. / BALL DIA.³ VS. SURFACE PRODUCED
3000 REV.
1200 REV.
300 REV.
MILL CHARGE
21000 G. - 1 3/8" + 1 1/4" BALLS
2800 G. CLINKER
TOTAL SURFACE PRODUCED SQ. CM. × 10³ PER 100 G.
RATIO OF CLINKER DIA. / BALL DIA.³ × 10¹²
0
20
40
60
80
100
120
2
3
4
6
8
10
20
30
40
60
80
100
200
300
400

Fig. 59

81. Discussion of Data. The data for this series is not complete because material was not available of proper size to give high ratios when used with the larger balls. Therefore, the conclusions drawn must be regarded as tentative until such time as more information is secured. However, the curves for the smallest ball size (Fig. 37) are reasonably consistent, and those for the larger sizes agree within the limits of the data available. They indicate that there is an optimum ratio of $\frac{\text{particle diameter}}{\text{ball diameter}^3}$ for which the grinding efficiency is a maximum, and that deviations in either direction from this figure cause marked decreases in the efficiency. This effect is not evident in the shortest runs but is very noticeable for the longer periods of grinding. The point of maximum efficiency is reached with particle sizes that are much largerthan are considered to be suitable in common grinding practice.

82. Conclusions. The tentative conclusions from this data are,

(1) The grinding efficiency of all sized balls acting on charges of varying particle sizes is nearly constant for very short grinding periods.

(2) For longer times of grinding there is an optimum ratio of $\frac{\text{particle diameter}}{\text{ball diameter}^3}$ for best efficiencies. Deviations from this ratio cause marked losses in efficiency.

83. Effect of this Ratio Upon theSize Distribution of the Product. There is some evidence to show that this ratio also influences the size distribution of the product. It is not complete enough to present here. In general, the tendency seems to be that the largest ratios produce the largest amounts of flour, while the small ratios yield a fairly uniform size distri-

bution.

V. Theoretical Amount of Energy Required For Grinding.

84. Current View. The view has been expresses many times(2) that a ball mill was actually a very inefficient machine. The reasoning followed in arriving at this conclusion was that the energy actually required to subdivide the material was very small, while the power expended was large. The energy required was assumed to be related to the heat of vaporization in that if it was ground to molecular size it would be a gas and the energy required to grind it to that stage of subdivision would be the heat of vaporization. Since the actual ground material was always very far from being reduced to molecular size, the energy required was only a small portion of the latent heat. Estimates have been made that the actual efficiency of a ball mill was only a fraction of one percent. The rest of the energy input was supposedly transformed into heat when the balls struck together.

85. Objections to This View. It would seem that, if these statements were true, it would be an easy matter to increase the efficiency of ball mills, with such a margin to work on. However, the longer the grinding experiments were carried on the more evidence was accumulated to show that it was very difficult to greatly affect the grinding efficiency. Moreover, such a definite relation as Rittenger's Law, which measures the surface produced in terms of the energy required, would hardly be expected to apply so accurately to the total energy input to the mill, if this amount is so large in comparison with the energy required. Also it is hard to conceive of so much energy being dissipated by the balls without being able to utilize more of it

by introducing morematerial between the balls. Because of these and similar reasons, the conviction has arisen that, while the energy required to actually cause division of the material might be small, there was some other demand for energy during the process which was connected with the material being ground and not with the ballsin the mill.

86. Possible Explanation of the LargeAmount ofEnergy Required for Grinding.

While seeking the explanation of this problem, an analagous case was thought about that may contain the answer to the question regarding the large demand for energy. Consider what happens when a piece of hardened steel is broken in a testing machine. As the load is applied the piece deforms elastically and stores up energy. This continues until the yield point is reached. This is also the breaking point,for this material . If, just before the breaking point, the process is reversed and the load slowly removed, it is possible to recover this energy that was stored in the elastic deformation of the piece. On the other hand, if the small additional load needed to raise the stress above the breaking point is added,the piece breaks. In this case none of the energy is recoverable, and it is transformed into heat through the hysteresis of the steel. It can be stated that the energy needed to separate the single piece of steel into two pieces was supplied by the last small increment of load added, and that this small amount would not appear as heat. It must be remembered,though, that the break would not have occured if the piece had not been stresses just to the breaking point before the last increment of load was added.

For this reason it would seem to be nearer the truth to say that the energy required was the entire amount needed to bring the piece to the breaking point. For brittle materials, this energy is equal to the modulus of resilience for aunit volume of material, or

$$\text{Energy} = \frac{S^2}{2\,E}$$

where S = stress at elastic limit

E = modulus of elasticity

If the same reasoning is applied to grinding, the energy required to cause a break would be equal to themodulus of resilience of of the material multiplied by its volume, and the energy required to grind a unit volume of material from one piece of unit dimension into a powder whose particles had dimensions of $\frac{1}{n}$ would be equal to the product of the modulus of resilience and the number of times the mass was stressed before it was reduced to the desired size. Since the energy required is proportional to the surface produced, it would appear reasonable to assume that the number of times the mass was stressed would likewise be proportional to the surface produced. The following is a numerical example of the abovereasoning.

Consider a one inch cube. If it is divided into four $\frac{1}{2}$" cubes the surface will be doubled, for the surface is proportional to $\frac{W}{D}$. To do this would require three breaks, one parallel to each pair of faces. The energy required would be $E_1 = \frac{3\,S^2}{2\,E}$. If the surface of the one inch cube is taken to be the unit of measure, this represents the energy required to produce unit surface. Now let the cube further divided until theparticles all have dimensions of 1/1000". The total surface will then be $\frac{1}{\frac{1}{1000}}$

or 1000 times the original, or the surface produced will be 999. The energy required would be 999 x $\frac{3\ S^2}{2\ E} = 1498.5\ \frac{S^2}{\cancel{2}\ E}$ Data found in Johnson's "Materials of Construction" gave the following values:

Mount Vernon Limestone.	Granite.
S = 7600 lbs. per sq.in.	S = 20,000 lbs. per sq.in
E = 2,800,000	E = 7,000,000
W = 139 lbs. per cu.ft.	W = 165 lbs. per cu.ft.

If these values are substituted in the above relation, and the results reduced to horsepower hours per ton, it would show that the limestone would require 28 hp hrs. and the granite 76 hp hrs to reduce one ton of material of one inch initial size to a product cosisting of particles 1/1000" in diameter.

This viewpoint is offered very tentatively, because it lacks any experimental confirmation. It is felt,however, that it is a more rational conception of the energy requirements of grinding than those previously expressed.

87. SUMMARY OF CONCLUSIONS.

Considering these grinding investigatins as a whole, the following are the principal conclusions.

(1) Rittenger's Law, that equal expenditures of energy produce equal increments of surface, is true for ball mills operating under given conditions of ball load and initial charge particle size.

(2) The grinding efficiency of ball mill is not affected by variations of the total amount of feed charge in the mill.

(3) The capacity of a mill is slightly decreased by running it with more charge than is necessary to fill the voids

between the balls. The efficiency does not decrease because the power input drops at the same time.

(4) When that part of the product passing the 200 mesh sieve is considered separately the following facts apply:

a. The larger the ratio of the initial particle size to ball size the larger the amount of very fine (-10 micron) material produced.
b. The smaller this ratio is the more uniform is the size gradation of the material produced.
c. Increasing the time of grinding does not change the above relations; it simply produces more material of approximately the same size gradation.

(5) The grinding efficiency depends upon the ratio of $\frac{\text{particle diameter}}{\text{ball diameter}^3}$. There is an optimum value for this ratio and deviations in either direction cause losses in efficiency.

(6) The tentative theory is offered that the amount of energy required for grinding a given material depends upon the modulus of resilience of that material and the size reduction desired.

It must be remembered that all of the conclusions presented here are derived from data obtained from runs made in a small, laboratory size, ball mill. Therefore these conclusions must be applied with great caution to large scale conditions, until additional experimental work has been completed for the purpose of correlating these facts with large scale operation.

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