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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

A NEW APPLICATION OF LIGHT TRANSMISSION IN FOLLOWING PARTICLE SIZE REDUCTION

I. APPLICATION TO COPPER PHTHALOCYANINE

DECEMBER 1950 - MARCH 1951

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I. INTRODUCTION

It is well known that the intensity of light passing through a medium is decreased if there are particles in that medium capable of scattering light. The more light that is scattered, the less will be the intensity of the light after it traverses the medium. It is also well known that the amount of scattering produced by the particles in dispersion is dependent not only on the concentration of particles but also on their size.

In the case of a dispersion of colored particles, the intensity of the transmitted light at a given wave-length is dependent not only on the particle size and concentration but also on the ability of the colored material to absorb light at that wave-length. A spectrophotometric transmission curve of such a dispersion will be the resultant of these effects and will change in shape as the particle size of the disperse medium is changed. This change in shape can be expressed in some manner to yield a particle size index. If the method of expression is such that it becomes independent of pigment concentration, it would enjoy a considerable advantage over methods requiring a measure of the concentration and would constitute a new approach to the problem of determining particle size.

This report describes such a method as applied to aqueous dispersions of CPC. It further describes a method for estimating the pigment concentration directly from a light transmission curve of a dispersion of the pigment. Work has also been done with other pigments in both aqueous and non-aqueous dispersionsindicating that the method is general in its scope. This additional work will be described in another report.

II. SUMMARY AND CONCLUSIONS

1. A particle size index of an aqueous dispersion of CPC can be had by obtaining the ratio of the optical density at the point of maximum light transmission to that at the point of minimum light transmission. The ratio so expressed will increase with increasing particle size.

2. This ratio is independent, within reasonable limits, of the concentration of the dispersion. Detailed directions for obtaining it is given in Appendix 1.

3. In an aqueous dispersion of CPC the light extinction coefficient at the wave-length of minimum light transmittance is a function of the particle size index. The smaller the particle size, the larger the extinction coefficient. This function can be determined by obtaining light transmission curves of dispersions over a particle size range of known concentration. With a knowledge of this function it becomes possible to obtain a value for the pigment concentration of a CPC dispersion directly from a light transmission curve. See Appendix 4 for detailed description.

4. An auxiliary light transmission cell holder for use on the G.E. spectrophotometer is described. This holder places the cells farther away from the integrating sphere. With the cells so placed, less scattered light finds its way into the integrating sphere during a light transmission measurement, minimizing the error so introduced.

III. THEORETICAL DISCUSSION

A. The Particle Size Index

In a pigmented system where a function of the pigment is to impart a color through selective light absorption, it is reasonable to consider that the particle size of the pigment is of that dimension where a decrease in size will give rise to a decrease in the amount of light scattering. If this were not so, i.e., if the pigment were of such large size that a decrease in size gives rise to an increase in light scattering, the pigment would be extremely poor from a tinctorial point of view. Its coloring power which is based on its ability to selectively absorb light as it passes through the particle would be very low.

If the foregoing is correct one would expect an increase in the intensity of the transmitted light as the particle size is decreased because there would be less light scattered. This is not the only thing that happens, however. In the case of a colored pigment, as the particle size decreases, the effective extinction co-efficient of the dispersion at the wave-length of maximum light absorption also increases. In other words, the dispersion of the colored pigment becomes a more efficient absorber of light at that wave-length where it normally is a good light absorber. There is one other observable effect on the curve shape as the particle size is reduced. The wave-length of maximum light transmission and minimum light transmission is shifted slightly in the direction of smaller wave-length. All three effects on the shape of the curve are illustrated for CPC.

To obtain a measure of something that correlates with particle size but at the same time is independent of a concentration measurement, one could use the shift of the curve to smaller wave-lengths but this shift is not very great and such an index would not allow the detection of small differences in particle size. The relative increase in transparency at the wave-length where the pigment is normally transparent compared to the relative decrease in transparency where the pigment is normally light absorbing is a more marked indication of change in particle size. If this change is expressed as a ratio of optical densities, the change will be independent of concentration, provided Beer's law holds at the wave-lengths where the optical density is measured. That this follows may be seen from the following considerations. Beer's law states that for a light path of constant length, the concentration is proportional to the optical density measured at a given wave-length.

$$D_1 = c\,k_1$$

D = optical density
c = concentration
k = extinction coefficient.

At a different wave-length the equation will have different values for d and k but the concentration will stay the same.

$$D_2 = c\,k_2$$

A ratio of optical densities will yield -

$$\frac{D_1}{D_2} = \frac{ck_1}{ck_2} = \frac{k_1}{k_2}$$

The concentration cancels out and the ratio becomes simply a ratio of extinction coefficients.

Experience with aqueous dispersions of CPC has shown that Beer's law does hold over a wide concentration range. It cannot be expected to hold over extreme concentration ranges because the degree of dispersion will be affected, multiple light scattering will feature to a greater extent with more concentrated dispersions and more scattered light will be included and measured with the transmitted light.

A particle size index can be defined by the following equation.

$$\text{Particle Size Index} = \frac{D_1}{D_2} \times 100$$

D_1 = optical density at wave-length of maximum light transmission.

D_2 = optical density at wave-length of minimum light transmission.

The factor 100 is introduced solely to bring the decimal point to a convenient place in the index. With the ratio taken in this manner, the smaller the ratio the smaller the average particle size.

B. Determining Pigment Concentrations

In the case of a colored solution, it is a simple matter to calculate the concentration of the colored constituent from a measure of its optical density if the extinction coefficient is known.

$$c = \frac{D}{k}$$

In the case of a colored dispersion this cannot be done because the extinction coefficient of the dispersion becomes a function of the particle size. If this function were known it would be possible to supply the proper extinction coefficient to the above equation and so calculate the concentration.

It is possible to establish this function by obtaining a series of dispersions representing a range in particle size. The pigment concentration for each dispersion must be obtained in some independent manner. Optical density measurements at the wave-length of maximum light absorption are obtained from light transmission curves. The extinction coefficient can then be calculated for each sample of the series. These values when plotted against the particle size index for each member of the series will yield a function from which it is possible to determine an extinction coefficient for any sample of known particle size index. Such a plot is illustrated in Figure 4 and its preparation will be described in detail in the experimental section, IV-D, and the detailed method for carrying out a determination is described in Appendix 4.

In using this method it is important that the type of dispersion to which it is applied be the same as the type used in obtaining the relationship between particle size index and extinction coefficient. This is necessary because no account is taken of particle size distribution or particle shape which conceivably can alter that relationship. Another limitation lies in the fact that for dispersions of large particle size, reproducibility becomes poor and the method becomes less reliable. In spite of these limitations, both the particle size index and the concentration estimation find practical application in following the course of particle size reduction during grinding of CPC.

IV. EXPERIMENTAL

A. Obtaining Spectrophotometric Curves

The G.E. self-recording spectrophotometer was used to obtain all curves. The transmission cell compartment of this instrument is so located with respect to the integrating sphere, that an appreciable amount of scattered light is also measured as transmitted light. This would give rise to an error. To minimize this error, a special transmission cell compartment was constructed allowing the placement of the transmission cells far enough away from the integrating sphere to reduce the amount of scattered light that enters the sphere. With this new position, it was assumed that the inclusion of scattered light in the measurement was sufficiently low to allow it to be neglected.

This cell compartment is constructed of steel shim stock 15 mils thick and is attached to the face plate holding the decentered lens and on that side of the plate facing the sphere. Two of the four screws holding the decentered lens mount are reversed enabling them to be used to screw on the cell holder. The dimensions of the cell holder are such that the conventional "Beckman" one centimeter light path cells conveniently fit in the holder side by side. Figure 2 is a picture of the cell holder by itself and also in position on the instrument with cells in place.

All transmission curves were made using this specially positioned cell holder.

B. Illustrating Validity of Beer's Law.

Since the success of expressing a particle size index in the manner described in Section III-A depends on the validity of Beer's law, it becomes desirable to test this validity as applied to the dispersions being measured. To do this , an aqueous dispersion of CPC was successively and quantitatively diluted. A light transmission curve was obtained for each of the dispersions. The initial concentration was designated as unit concentration and subsequent concentrations were calculated in terms of unit concentration. For example, the initial dispersion would be given an arbitrary concentration of one. A one to one dilution would then have a concentration of 0.5, a one to two dilution would have a concentration of 0.333, etc.

Optical densities were obtained at the wave-length of minimum light transmittance and maximum light transmittance. These optical densities were then divided by the relative concentration of each dispersion giving the optical density per unit concentration. This is essentially a measure of the extinction coefficient in terms of the arbitrary concentration unit and should be a constant if Beer's law holds. Table I shows the results of such an experiment.

TABLE I

CPC DISPERSION - OPTICAL DENSITY

Dispersion No.	Relative Concentration	Optical Density ÷ Relative Concentration: At λ of Min. light Trans.	Optical Density ÷ Relative Concentration: At λ of Max. light Trans.	*Particle Size Index
1	1.000	1.40	0.353	25.2
2	0.500	1.58	0.369	23.4
3	0.333	1.56	0.366	23.4
4	0.250	1.58	0.364	23.1
5	0.200	1.58	0.365	23.1

*$\frac{\text{Optical Density at } \lambda \text{ of Max. light transmittance}}{\text{Optical Density at } \lambda \text{ of Min. light transmittance}} \times 100 =$ Particle Size Index

By reference to Table I it is seen that with the exception of No. 1, which is the most concentrated dispersion, the ratio of optical density to concentration is quite constant at each wave-length. It is also seen that with the exception of dispersion No. 1 the particle size indices calculated from all of the curves are about the same. Dispersion No. 1 has an optical density at the wave-length of minimum transmittance of 1.40. This means a percent transmittancy of 4%. To assure acceptable conformity to Beer's law, the percent transmittancy

should not be allowed to get as low as 4% for this particular dispersion. Ten percent is a safe lower mimit and only slight error is introduced at transmittancies as low as 5%. For dispersions of smaller particle size there will be less light scattering and transmittancies as low as 4% and even lower can be tolerated. This gives one a wide convenient latitude in concentration and even if there is some doubt as to the validity of applying Beer's law to some particular dispersion, it can be easily checked by making successive dilutions.

C. Illustrating Determination of Particle Size Index

A series of grinds in which the variable is time of grinding is ideally suited to illustrate this application. CPC blue and also the green polychlor CPC have been used but only its application to Blue CPC will be illustrated. The sample used for illustration is a chlorine-free alpha phase CPC, 1383-88-D, and the grinding was accomplished by "HF" milling an aqueous slurry of the pigment. The aqueous slurry contained an indefinite amount of borax and approximately 60% "Blancol" on the pigment basis. "HF" milling is a special type of milling producing a high shearing force through high energy input stirring.

The slurry so obtained is far too concentrated to obtain a light transmission curve. Dilution is carried out by adding a drop or two of the slurry to about 30 ml of a 0.1% "Blancol" solution. With experience, visual inspection of the resulting dispersion shows when the concentration is about right for the light transmission measurement. A 0.1% "Blancol" solution is used in the comparison cell to cancel out the slight color of the "Blancol" solution. After concentration adjustment so that the point of minimum transmission is not below 5% and preferably around 10%, a curve is obtained following the method described in IV-A. Figure 3 shows the type of curve obtained when the particle size index is plotted against time of grinding. A smooth curve results which shows that the method is well adapted to serve as a testing method for following the course of particle size reduction during grinding. Figure 1 shows the type of light transmission curves obtained from which the particle size index is calculated. Appendix 1 gives detailed directions for determining the particle size index.

D. Illustrating Determination of Pigment Concentration

The underlying principles are discussed in Section III-B. For its illustration acid-pasted chlorine-free CPC will be used. First, it is necessary to know the relationship between particle size index and extinction coefficient. This is accomplished by obtaining a series of dispersions of varying particle size. In this case the series was obtained by varying the time of grinding.

Laboratory grinds were made using the "HF" milling technique. The charge was 60 grams of pigment, 38 grams of "Blancol" and 600 grams of Borax. The grinding times were 1 hr., 2 hrs., 3 hrs., 4 hrs.; the samples were labeled 1383-88A, B, C, D, respectively. The dry pigment under these conditions contained about 30% CPC. A 0.025 g. sample of

the dry pigment was dispersed in a 0.1% "Blancol" solution. The volume was made to 250 ml. A 25 ml portion of this dispersion was further diluted to 50 ml. A 0.1% "Blancol" solution was used for all dilutions. At this dilution, the transmittancy at the point of maximum light absorption was around 10%. Transmission curves were obtained of the series and the particle size index calculated using the formula:

$$\text{Particle Size Index} = \frac{\text{Optical Density at Wave-length of Max. light transmittance}}{\text{Optical Density at Wave-length of Min. light transmittance}} \times 100$$

The next step was to determine the percent CPC actually present in the dispersion. Any method of analysis for CPC would be suitable for this purpose. For example, a copper analysis on the dry pigment calculated to CPC could be used. In this case, however, light transmission measurements in the near infra-red of sulfuric acid solutions of the pigment in the dispersion was used. This method has the advantage of making the analysis directly on the dispersed system used for the light transmission measurements. This makes it unnecessary to precisely know the initial weight of pigment used. This method suffers from the disadvantage, however, of requiring a knowledge of the extinction coefficient of pure CPC in sulfuric acid. Actually such a knowledge is necessary only if an absolute measure of CPC concentration is required. Even without knowing the true extinction coefficient, valid relative measurements can be made, using as a reference, some reasonably pure sample of CPC.

For this experiment, such a sample was used as a reference and its treatment is described in Appendix 2. The method of analysis of aqueous dispersions of CPC using the method which involves solution in H_2SO_4 is given in Appendix 3.

With a knowlege of the concentration of CPC in the aqueous dispersion, it becomes possible to calculate the effective extinction coefficient "k" of the dispersion from the equation:

$$k = \frac{D}{c}$$

D = Optical density at wave-length of Min. light transmittancy
c = Concentration in g/l.

It will be seen that "k" for aqueous dispersions will increase with decrease in particle size index. Figure 4 shows a plot of particle size index against extinction coefficient of the aqueous dispersion at the wave-length of maximum light absorption.

It now becomes possible to determine the concentration of a dispersion obtained in a manner similar to the dispersions used to obtain the plot of Figure 4 by a direct light transmittancy measurement of the aqueous dispersion. No solution in H_2SO_4 is required. This determination is described fully in Appendix 4.

APPENDIX 1

Determination of Particle Size Index of CPC Aqueous Dispersions

The pigment must be dispersed in some medium and in such a manner that the pigment does not flocculate. A 0.1% aqueous solution of "Blancol" serves well for aqueous grinds of CPC.

Adjust the pigment concentration of the dispersion by diluting with 0.1% "Blancol" until, by visual inspection, it appears that the minimum percent transmittance will be no less than 4% and preferably around 10%. Place some of this diluted dispersion in a one cm. light path cell. Place some 0.1% "Blancol" solution in another one cm. light path cell. This solution should be from the same bottle as that used in diluting the original CPC dispersion.

Place both cells in the special cell compartment on the G.E. spectrophotometer. This specially constructed cell compartment is described in section IV-A and shown in Fig. 2. Obtain a spectrophotometric light transmission curve.

Make two transmittancy measurements, either from the curve or directly from the instrument. One measurement, "T max." is made at the point of maximum light transmittance and the other "T min." is made at the point of minimum light transmittance. Calculate the optical density "D" for each of the light transmittance measurements from the formulas:

$$D_1 = \log \text{ T max.}$$

$$D_2 = \log \text{ T min.}$$

Calculate particle size index from the formula.

$$\text{Particle Size Index} = \frac{D_1}{D_2} \times 100$$

APPENDIX 2

Determination of Extinction Coefficient of Copper Phthalocyanine in Sulfuric Acid

I. SAMPLE PREPARATION

A. Acid-Pasted Chlorine-Free CPC

A special CPC sample labeled 1226-7A was used. This sample had been specially purified by thorough acid-pasting and exhaustive washing. It was analyzed for copper using KCR 159. This yielded 10.89% Cu. A molecular weight of 576.1 yields a conversion factor of 9.067, this makes the purity 98.6% CPC.

B. LB/CPC

A special LB sample labeled 1226-7B was used. This sample had been purified in a manner similar to 1226-7A. A copper analysis by KCR 159 yielded 10.49% Cu. A molecular weight of 599.9 yields a conversion factor of 9.44. This makes the purity 99.0% CPC.

II. PROCEDURE

Weight 0.1000 g. dry pigment into a tared 50 ml beaker on an analytical balance. Add approximately 30 ml of concentrated CP sulfuric acid to dissolve CPC. Pour solution into a tared 70 ml weight burette. Wash out beaker 3 or 4 times with sulfuric acid and add to weight burette to a total volume of about 65 ml or 100 g. net weight. Weigh burette and contents before shaking (to avoid leakage at stopper!). After determining total weight, shake contents thoroughly to assure a uniform solution. If some sulfuric acid solution oozes past the stopper, it must be wiped off first with a damp cloth and finally with a dry cloth. There should be no drift in the weight due to moisture pickup of the sulfuric acid. Reweigh the burette and contents. Draw off about 20 drops into a clean 250 ml. volumetric flask and reweigh weight burette and contents. The difference in weight represents the weight of aliquot taken for a determination. Add sulfuric acid to flask and fill to mark. Shake flask and contents thoroughly to assure a uniform solution.

Determine the percent transmittance of this solution on the Beckman spectrophotometer at the two minimum points found at about 700 and 790 mμ. This is done by pouring some of the sulfuric acid solution from the 250 ml flask into a dry 1 cm. cell and some CP sulfuric acid into a second dry cell to be used as a blank. This sulfuric acid should be from the same bottle as that used in making up the solutions. Due to a possible instrumental wavelength error the actual wave-lengths at minimum transmittancy must be determined by measurement at various wave-lengths in the vicinity of 700 and 790 mμ.

Calculations:

Convert transmittance measurements to optical density measurements using the formula Optical Density = -log Transmittance.

Calculate the Extinction Coefficient "k" using the following formula:

$$\frac{\text{Optical Density at min. \% Transmittance}}{\text{Dry sample weight (grams)}} \times \frac{\text{Weight of aliquot (grams)}}{\text{Total weight of solution (grams)}} \times \frac{1}{\text{Volume of flask (liters)}} = \text{Extinction Coefficient, "k" (Sample basis)}$$

Sample Calculation

$$\frac{0.943}{\frac{0.1000}{106.6} \times \frac{0.6180}{0.250}} = 407 \text{ (on sample basis)}$$

Replicate determinations of "k" look as follows:

	Cl-free CPC		LB/CPC	
	λ = 700 mμ	λ = 790 m	λ = 700 mμ	λ = 795 mμ
	59.3	407	57.9	375
	58.2	396	55.3	369
	59.0	407	54.8	360
	60.0	—	56.6	371
Ave.	59.1	403	56.1	369

Since it is known that the samples used were not 100% CPC, the average extinction coefficient on the sample basis is converted to the 100% CPC basis using the % CPC based on the Cu analysis. Following are the calculations to give the 100% CPC basis extinction coefficients.

Cl-free CPC

% CPC = 98.60%

$$\frac{59.1}{0.986} = 59.94 = k_{800} \text{ (100\% CPC basis)}$$

$$\frac{403}{0.986} = 408.6 = k_{790} \text{ (100\% CPC basis)}$$

LB/CPC

% CPC = 99.03%

$\frac{56.1}{0.9903} = 56.7 = k_{700}$ (100% CPC basis)

$\frac{369}{0.9903} = 373.0 = k_{795}$ (100% CPC basis)

NOTE: For all extinction coefficient measurements the unit light path is one centimeter and the concentration is expressed in grams per liter.

APPENDIX 3

Determination of CPC Concentration in an Aqueous Dispersion by Light Transmission of Sulfuric Acid Solution

The aqueous dispersions to which this method has been applied have contained "Blancol", borax and dextrin. Light absorption of these ingredients in the near infra-red was slight at the concentrations used but, even so, a correction was applied assuming that the light absorption of these ingredients was the same at the two-wave-lengths chosen to measure CPC. Actual curves showed the justification of this assumption.

Procedure

The dispersion should contain CPC at a concentration of about 0.03 g./l. If the concentration is much higher than this, as it usually is, a quantitative dilution should be made to approximate this concentration.

Precisely pipette 2 ml of a dispersion containing approximately 0.03 g. CPC/l. into a 50 ml. beaker. Evaporate to dryness. Take up with a few ml. concentrated H_2SO_4. Work with a stirring rod making sure all the pigment is dissolved. Transfer to a 25 ml. volumetric flask by repeated additions of H_2SO_4 until complete transfer of the CPC to the flask has been accomplished. Make to the mark with more H_2SO_4. Shake thoroughly. The H_2SO_4 will tend to char the dextrin but this action will not greatly affect the near infra-red transmittance.

Determine the percent transmittance of this solution on the Beckman spectrophotometer at the two minimum points found at around 700 mμ and 790 mμ. This is done by pouring some of the sulfuric acid solution into a dry one cm cell and some CP sulfuric acid into a second dry cell to be used as a blank. This sulfuric acid should be from the same bottle as that used in making up the solutions.

Calculation

This calculation corrects for the effect of "Blancol" and Dextrin on the light transmittance measurement. The correction assumes that the influence of the "Blancol" and dextrin is the same at 700 mμ as it is at 790 mμ.

The following terms are defined as follows:

c = Concentration (grams/liter)

D_1 = Optical Density of the solution measured at 700 mμ

D_2 = " " " " " " at 790 mμ

k_1 = Extinction coefficient of CPC at 700 mμ

k_2 = " " " at 790 mμ

$r = k_2/k_1$

$\triangle D$ = The correction to be substracted from D_1 or D_2 to compensate for the effect of "Blancol" and dextrin. It is assumed that the same correction will apply to both D_1 and D_2.

With the foregoing assumption, the following equation is true.

1. $$\frac{D_2 - \triangle D}{D_1 - \triangle D} = r$$

or expressing in terms of $\triangle D$

2. $$\triangle D = \frac{r\ D_1 - D_2}{r - 1}$$

3. $$D_2 \text{ (corr.)} = D_2 - \triangle D$$

4. $$c = \frac{D_2 \text{ (corr.)}}{k_2}$$

Sample Calculation

$D_1 = 0.172$

$D_2 = 1.113$

$k_2 = 408.6$

$r = 6.82$

From equation (2)

$$\triangle D = \frac{6.82 \times 0.172 - 1.113}{6.82-1} = 0.010$$

From equation (3)

D_2 (corr.) = 1.113 − 0.010 = 1.103

From equation (4)

$$c = \frac{1.103}{408.6} = 0.002702 \text{ g./liter.}$$

APPNNDIX 4

Determination of CPC Concentration in Aqueous Dispersion by Light Transmission of Aqueous Dispersion

I. PREPARATION OF WORKING CURVE

It is first necessary to prepare a working curve similar to that illustrated in Fig. 4. To do this, proceed as follows:

1. Obtain a series of dispersions of various average particle sizes, i.e., by varying the time of grinding or by particle size classification of some sort.

2. Obtain light transmission curves over the visible portion of the spectrum of quantitative dilution made from these dispersions. For aqueous dispersion, 0.1% "Blancol" has proven suitable. The dilution should be of such magnitude that the minimum light transmittance is about 10% for an optical light path of 1 cm. The method of measuring the transmitted light should be such that the amount of scattered light, measured along with the transmitted light, is kept at a minimum. This can be accomplished on the G.E. spectrophotometer by fashioning a special cell holder shown in Fig. 2 and described in section IV-A.

3. Calculate particle size indices from the light transmission curves using the formula:

$$\text{Particle Size Index} = \frac{\text{Optical Density at wave-length of Max. Light Trans.}}{\text{Optical Density at wave-length of Min. Light Trans.}} \times 1($$

4. Obtain pigment concentration "c" of the dispersion used for the light transmission curves by using the method described in Appendix 2 or some other suitable method. It is not necessary that the analysis be performed on the same dispersion used to obtain the light transmission curve. Some other dilution of that dispersion may be used, but in that case, it must be possible to calculate the concentration of the actual dispersion used in obtaining the light transmittance measurements by applying the proper dilution factor.

5. From a knowledge of the concentration of the dispersion obtained in step 4, calculate the extinction coefficient "k" at minimum light transmittancy of the dispersion using the optical density previously obtained for the denominator in the equation of step 3. To calculate this extinction coefficient "k", use the formula:

$$k = \frac{\text{Optical Density at wave-length of min. light transmittance}}{\text{Pigment concentration (g/l)}}$$

6. Plot "k" obtained from step 5 against "c" obtained from step 4. The plot will have the general appearance of that illustrated in Fig. 4. This is the "Working Curve".

II. USE OF WORKING CURVE TO DETERMINE CONCENTRATION

Note

The dispersion to which this method is applied should be one derived in a manner similar to those used in preparing the "Working Curve".

1. Quantitatively adjust the concentration of the dispersion to a point, where the light transmittance is about 10% for a 1 cm light path. Obtain a light transmission curve and calculate the particle size index from the equation:

$$\text{Particle Size Index} = \frac{\text{Optical Density at wave-length of Max. light trans.}}{\text{Optical Density at wave-length of Min. light trans.}} \times 100$$

2. From the "Working Curve" determine the "k" value which corresponds to the "Particle Size Index" calculated in step 1.

From the optical density at wave-length of minimum light transmittance "D" as determined in step 1, calculate pigment concentration from the formula:

$$\text{Pigment Concentration} = \frac{D}{k}$$

This will give the pigment concentration of the dispersion from which the light transmission curve was obtained. If this represents a dilution, which it usually does, the concentration of the original dispersion is obtained by multiplying by the proper dilution factor.

III. SAMPLE CALCULATION

Data

Pigment dispersion diluted from 25 ml to 50 ml.

% Transmittancy "T" Maximum ---------- 90.7
" " "T" Minimum ---------- 11.5

Calculation of Optical Density

$D = -\log T$

$D_1 = -\log 0.907 = 0.0424$ ("T" maximum)

$D_2 = -\log 0.115 = 0.939$ ("T" minimum)

Calculation of Particle Size Index

$$\text{Particle Size Index} = \frac{0.0424}{0.939} \times 100 = 4.52$$

Calculation of Concentration

From reference to graph of Fig. 4 a particle size index of 4.52 corresponds to a "k" of 55.5.

Concentration of diluted dispersion = $\frac{0.939}{55.5}$ = 0.01693 g/l

Concentration of original dispersion = 0.01693 x 2 = 0.03396 g/l

Figure 1. Aqueous Dispersions of CPC Varying Grinding Time

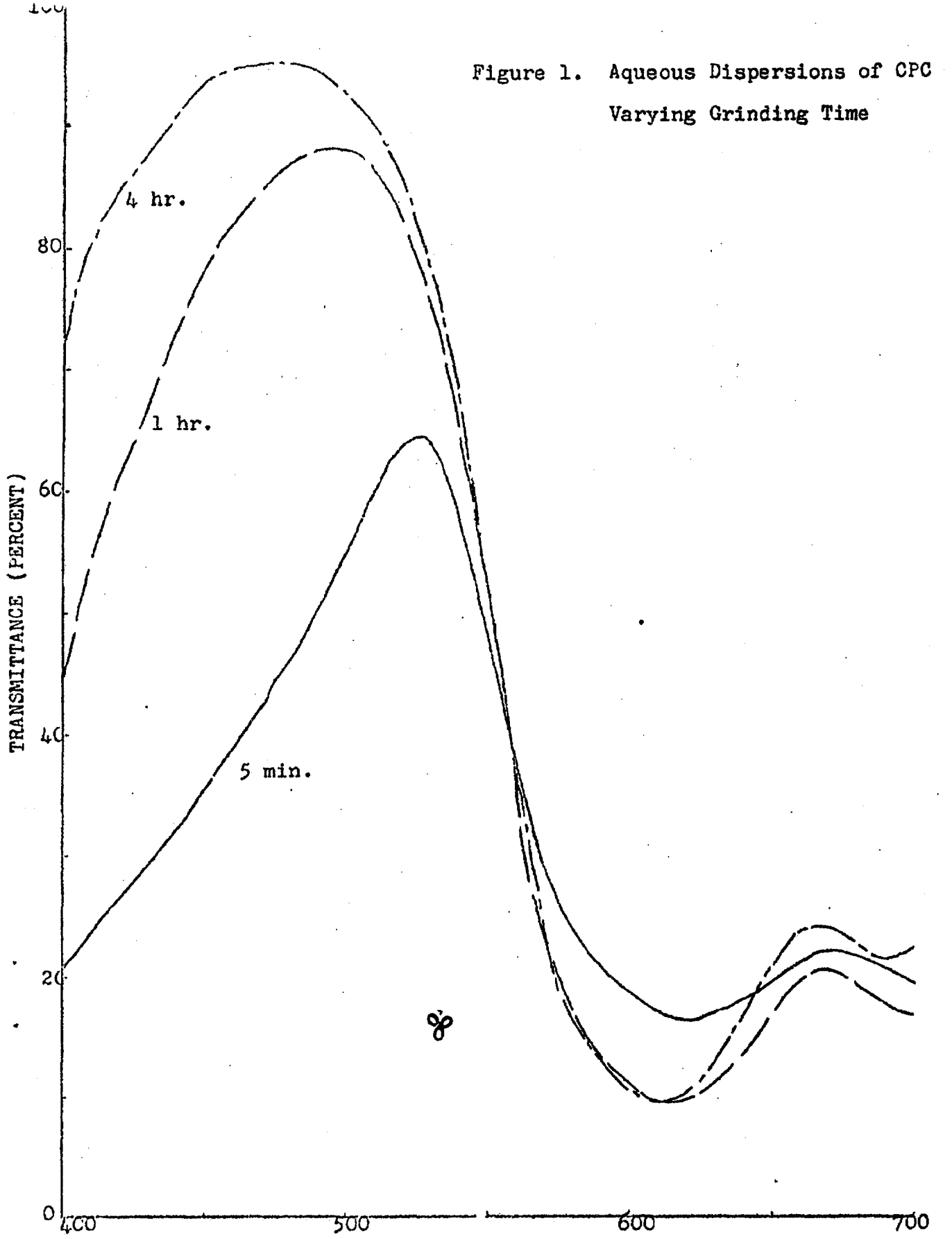

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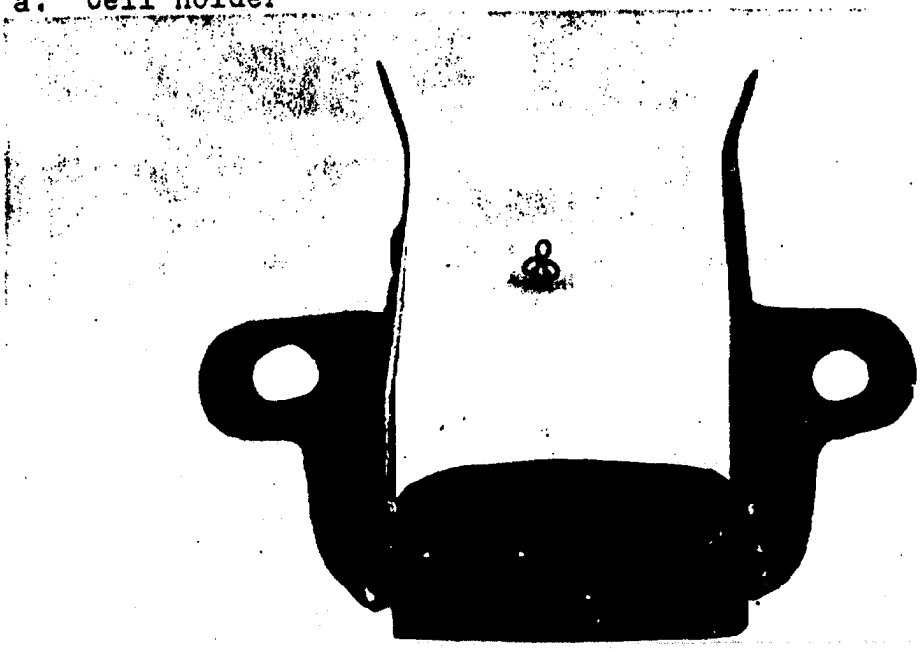
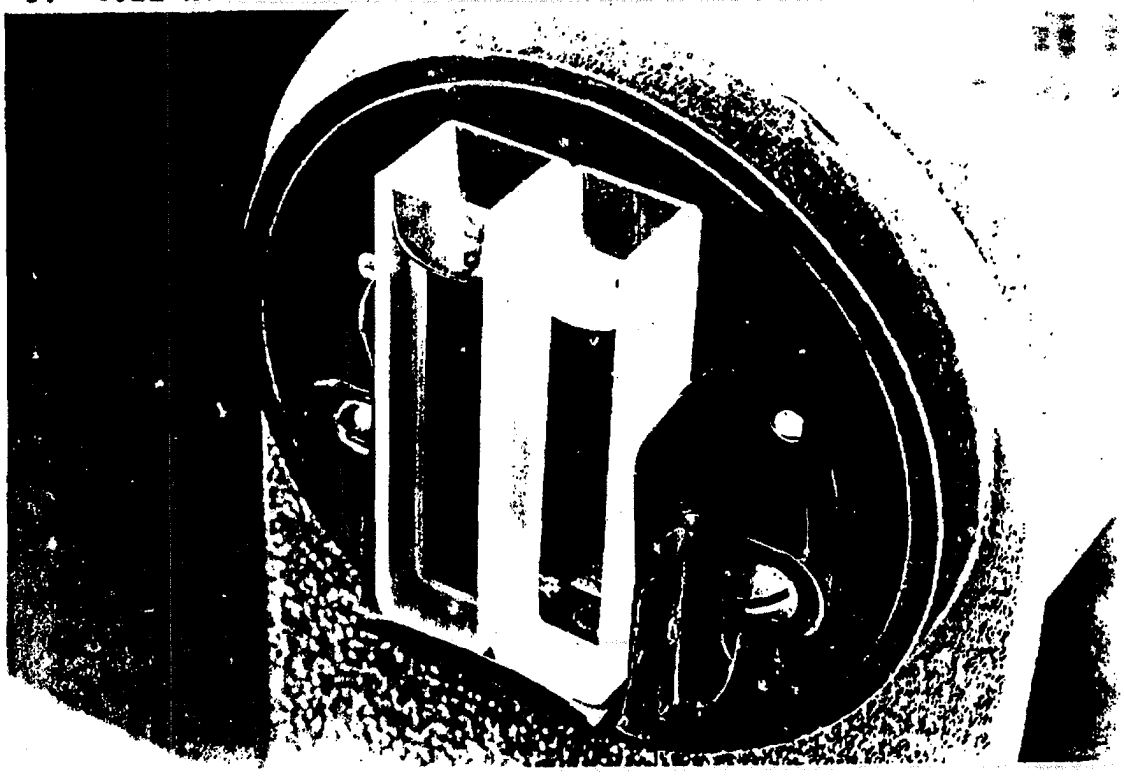
Figure 2.

a. Cell holder



b. Cell holder at decentered lens.



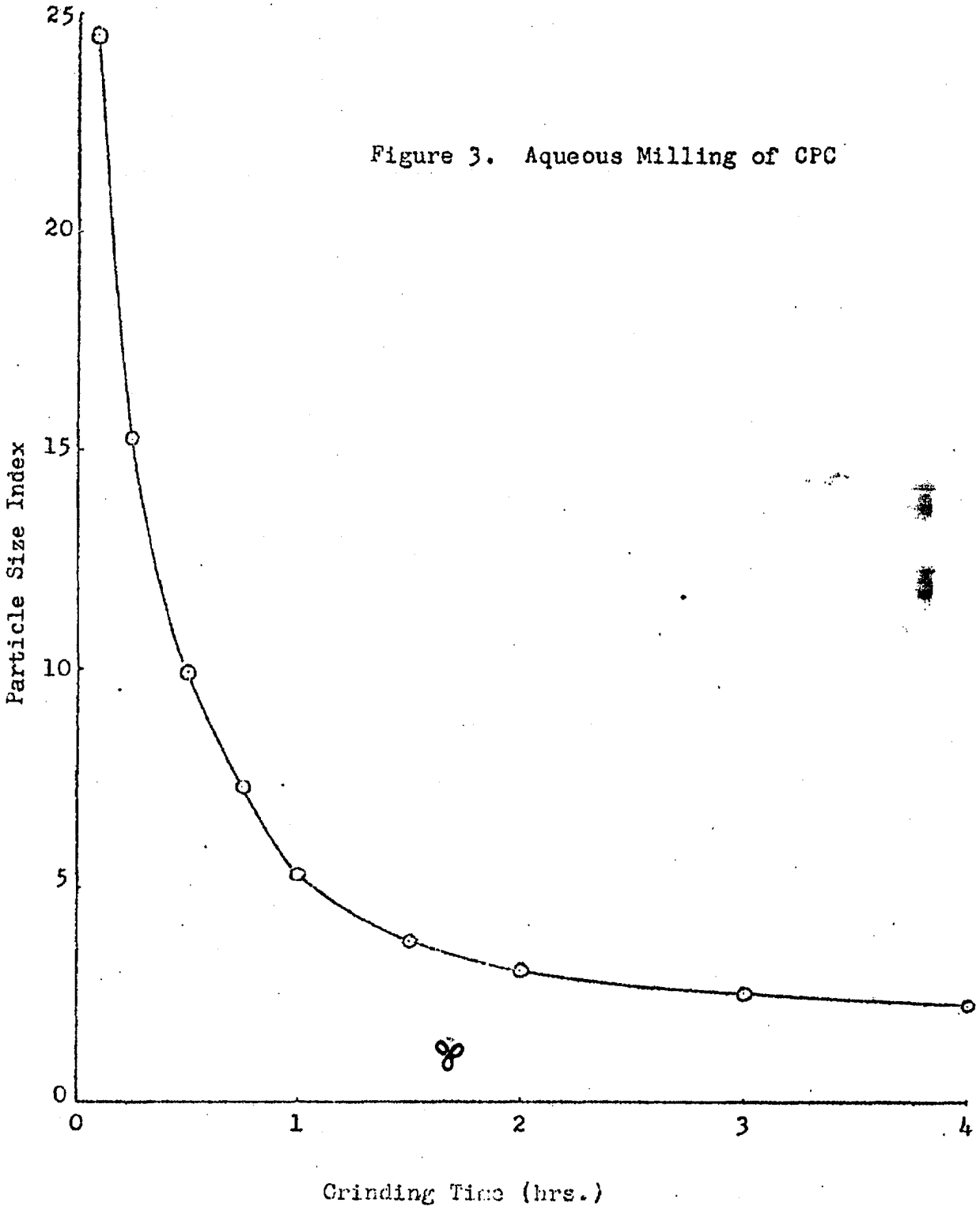


Figure 3. Aqueous Milling of CPC

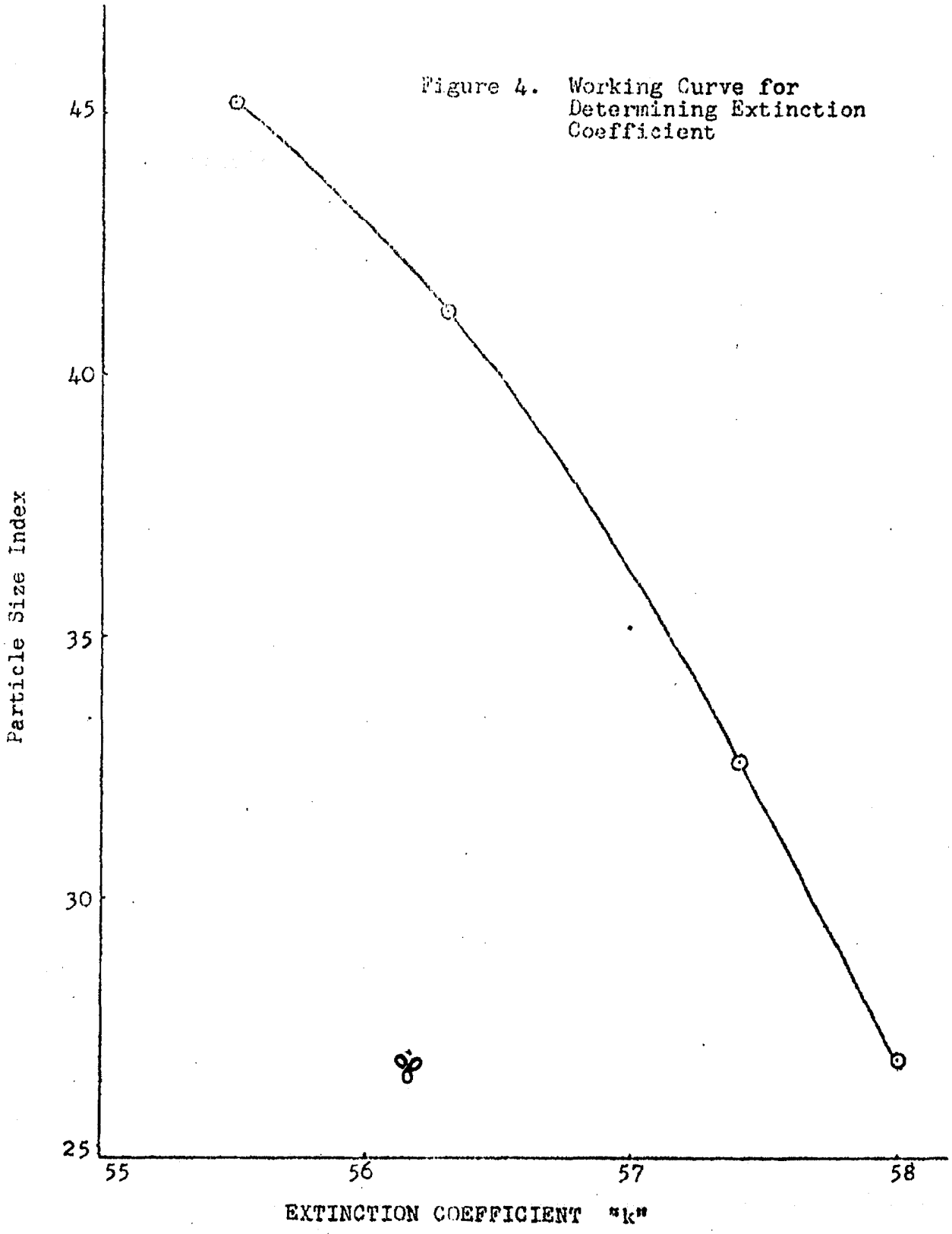


Figure 4. Working Curve for Determining Extinction Coefficient