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INVESTIGATIONS INTO METHODS OF
PHOTOMETRIC MEASUREMENT OF SURFACE AREA AND
AVERAGE DIAMETERS OF FINE PARTICLES

Head

by

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ABSTRACT

The problem of surface area and particle size determination has been examined. A survey of the methods for measuring the surface areas and average sizes of aggregates of fine particles was made and a method selected for improvement.

An apparatus employing the principles of fine particle measurement established in turbidimetry was constructed. Experimental work with the apparatus was conducted in an attempt to establish a single method of particle size measurement.

The results warrant the conclusion that the apparatus will measure average surface diameters of single component materials, constant assaying materials, and mixtures containing one semi-transparent material. A value for the relative surface area of these materials can also be determined.

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I - INTRODUCTION

The measurement of finely divided material is a problem for which there has been no single general solution. Particulate material can have such varied physical characteristics that it is doubtful if any single method of measuring aggregates of particles will ever be developed.

In the selection of a method for particle size measurement, the type of material, the size range of the material, and the intended use of the measurement must be considered.

Insofar as the selection is affected by the type of material, particle shape is the most important physical characteristic to be considered. Particle size is not an absolute term but must be defined in relation to the regularity of particle shape. In order that aggregates of particles can be treated statistically it is necessary that irregular shapes be considered as equivalent geometric shapes. The equivalent particle diameter, which is generally referred to as the particle diameter, is considered as the diameter of a hypothetical sphere or circle which has at least one physical characteristic equal to the corresponding characteristic of the related particle. It is readily seen that as particle diameters based on the length, surface, or volume of a particle need not have the same numerical value the inherent particle shape of a material must be considered before a method of particle size

measurement can be selected.

Two methods of particle size measurement that are directly dependent on particle shape illustrate the importance of this characteristic. If, for instance, a mixture of two types of materials, one having a compact, granular form and the other a needle-like structure, were screened in an attempt to determine the size distribution, the results would have little meaning as the needle-like structure of the one material would prevent it from passing through interstices apparently larger than those necessary. On the other hand if sedimentation rates were the sole basis for measurement, size determinations of materials in which the particle form varied greatly and erratically from the spherical form could not be reliable. Methods involving sedimentation are dependent on the use of Stoke's law which states that the diameter of a particle is proportional to the square root of the rate of settling. Stoke's law, however, holds true only when the particles dealt with are spheres and can be used only as an approximation even when the degree of deviation from sphericity is constant.

The electrical, magnetic, and optical properties, and the specific gravity of a material must also be considered before a method of particle size measurement is selected.

An equally important factor in the selection of a suitable method of particle size determination is the size range of the material to be examined. The field of fine particle technology is generally considered to deal with particles of which the upper size limit is 10^4 microns and the lower size limit is 10^{-1} microns. Screening procedures are fairly satisfactory for size measurements of particles between the limits of 10^4 and 50 microns. Sedimentation, microscopy,

turbidimetry and elutriation are accepted methods for size determination in the narrow range from 1 to 50 microns. For particles that are in the size range below 1 micron the centrifuge and the electron microscope are widely used to determine size-distributions.

The above methods of size determination are not completely restricted to the size ranges given as it is often possible to obtain experimental results in one size range and extrapolate these results, by means of smooth curves or other acceptable mathematical procedures, into the ranges bounding the initial range. However it is apparent that the complete size range with which particle size technology is concerned, is too large to be encompassed by one single general method of particle size measurement.

Finally, it is essential that any method for determining particle size be chosen with some regard for the intended use of the measurement. It has already been indicated that a number of values could be obtained for the equivalent particle diameter depending on whether it was determined on a length, area, or volume basis. If it is desired to obtain a particle diameter that is a measure of the surface then a method should be used which determines particle size by measuring particle surface. Similarly, if it is desired to obtain a particle diameter that is a measure of the volume then a method should be used that determines particle size by measuring particle volume. There are two other methods of arriving at equivalent particle diameters, each method giving an equivalent particle diameter expressed in terms that will serve a specific purpose. One of these methods is dependent on a volume-surface basis and the other on a weight basis.

Most methods of particle size measurement, including sedimentation, elutriation, and centrifuging, give equivalent particle diameters as a function of volume. Microscopy may give equivalent diameter as a function of length or area. Turbidimetry gives the equivalent diameter as a function of area.

If it is desired to obtain a value for specific surface of a material it is not necessary to be concerned with particle diameter. Methods involving solubility and gas adsorption are the best means for determining specific area but they give no indication of the size-distribution. However, in studying rates of oxidation and solution, catalysis, adsorption, crushing and grinding, covering power and other similar operations, specific surface is the prime criterion, whereas, particle ~~shape~~^{size} is relatively unimportant. The reverse is true in the study of ceramics, pigments, abrasives, cements, and other similar fields.

It is thus apparent that a great many factors must be considered before a method of determining average particle size, size-distribution, or specific surface area can be selected. The problem of particle size measurement, in general, can become very complex if the materials dealt with have indefinite and abnormal physical properties and if the range of sizes encountered is large.

A method of particle size measurement that is rapid, accurate and suitable for a wide range of materials and sizes, is desired in the research laboratory and in industry. The method should be simple and, therefore, not require the services of specially trained technicians. It should be capable of determining all types of average particle sizes, size-distributions, and if possible, specific surface area. As suggested previously, it is unlikely that a method of particle size measurement

could fulfill all of these requisites. However, the methods in use at present can be improved and extended so that they may be applicable to an ever widening range of conditions.

A survey of some of the disadvantages of the common methods of particle size determination indicates where improvement is necessary.

Sedimentation, elutriation and the ordinary turbidimetry methods of particle size determination are dependent on the laws of settling hypothesized for truly spherical particles. Particle shape is often involved in an indeterminate manner and thus affects the results. Further, these methods tend to be tedious and require considerable knowledge and skill.

Particle size determination by microscopy is tedious and exacting, and it is often impractical to examine more than a very minute part of the whole sample.

Centrifuging has the great disadvantage that it is applicable only to the sub-micron particle size range.

Solution and adsorption methods of surface area determination are time consuming and require considerable apparatus and skill.

Permeability methods of surface area determination are greatly affected by particle shape and are consequently subject to serious error.

As it would appear that the best approach to the problem of improving the methods of fine particle measurement would be the development of a method which inherently measures surface area but which gives results capable of being interpreted as average particle size, and as solubility, adsorption, permeability and turbidimetry methods meet the above specifications, turbidimetry was selected for initial study since it appeared the simplest of the four.

An apparatus that would measure, with a high degree of accuracy,

the surface area of the powders of a few of the simple minerals has been described by John W. Bell ¹⁾. His apparatus consisted, essentially, of a photometric device that would measure the turbidity of fine particle suspensions. Turbidity, in this case, was considered as a function of surface area.

Bell's method of surface area determination appeared to be suitable for extension and improvement so that it might approach a single general method of particle size measurement.

II - PRACTICAL AND THEORETICAL CONSIDERATIONS

OF THE PHOTOMETRIC METHOD

Bell's method of surface area measurement by turbidity determination is a modification of the method evolved by Wagner ²⁾ in 1933. In Wagner's device a suspension of particles is placed in a glass sedimentation tube, 8 inches in depth and $1\frac{1}{2}$ inches square, and stirred. At a fixed distance from the tube is placed a standard lamp which passes a beam of light through the suspension. The light absorbed is indicated on the other side of the tube by means of a photoelectric cell. The standard lamp is provided with a condenser to remove radiant heat. Wagner used Stoke's law to determine the size-distribution of particles at any level in the settling tube. The general equation fitting the observed data was

$$S_d = c(\log I_0 - \log I_d) = c(2 - \log I_d)$$

where S_d is the surface area of all particles in the sample smaller than d , c a constant for the material observed, I_0 the strength of the incident light, and I_d the intensity of the light in microamperes transmitted

through the suspension at the end of the time interval required for a particle d in diameter to settle from the surface of the suspension to the center line of the light beam. After a standard curve has been derived, only a single reading is required for determination of the surface of particles less than a stated size. Integration over all sizes may be accomplished easily. Normally the Wagner method can be considered only as a means of obtaining a relative surface and the surface measured depends upon the standard used and the accuracy with which the surface was originally determined. According to Dalla Valla³⁾, the method is in no sense absolute, nor can it be applied safely to different materials unless suitable standards are available for each. Moreover, the accuracy of the method is affected by color, adsorption, birefringence, refraction, and reflectivity which are involved in an indeterminate manner.

Bell's apparatus, in its barest elements, consisted of two photo-electric cells, a standard light source, and a circulating chamber for fine particle suspensions. The reciprocal of the weight of material in suspension, necessary to cut off a certain amount of light in front of one of the photo-electric cells was considered proportional to the specific surface of that material. The amount of light to be cut off was kept standard by upsetting the balance of the two photo-electric cells a certain amount and then bringing them into balance again by the addition of material to the circulating chamber. A schematic diagram of Bell's apparatus is given in Figure 1.

Bell's method differs from the Wagner method in that turbidity determinations are made on a well mixed suspension and consequently settling equations involving Stoke's law are not involved.

However, the method is still dependent on the optical properties of the materials under consideration. It is necessary, therefore, to determine whether or not these properties affect the results appreciably and, if they do, to determine whether they remain constant for any one material down through the size ranges concerned.

It is a well known fact that decrease in particle size of most materials will change their color sensed by the human eye. However, if these materials are examined under a microscope it will be noted that even the smallest particles retain the color and lustre which is evident to the eye at larger grain size. Although this color change is readily apparent to the human eye it is difficult to state in what manner it would affect the operation of a photoelectric device.

Reference to the literature, 4, 5, 6,) on the subject of reflectivity with respect to fine particles, indicates that even with quartz the degree of maximum light reflection should be small enough to make the change of light reflection with decrease in particle size a negligible factor.

According to the Fresnel equation

$$R = \frac{(n_1 - n_0)^2}{(n_1 + n_0)^2} \quad \text{EQUATION 1}$$

where

R is the reflection coefficient or
fractional part of the incident light
that is reflected,

n_1 is the refractive index of the solid,

and

n_0 is the refractive index of the medium.

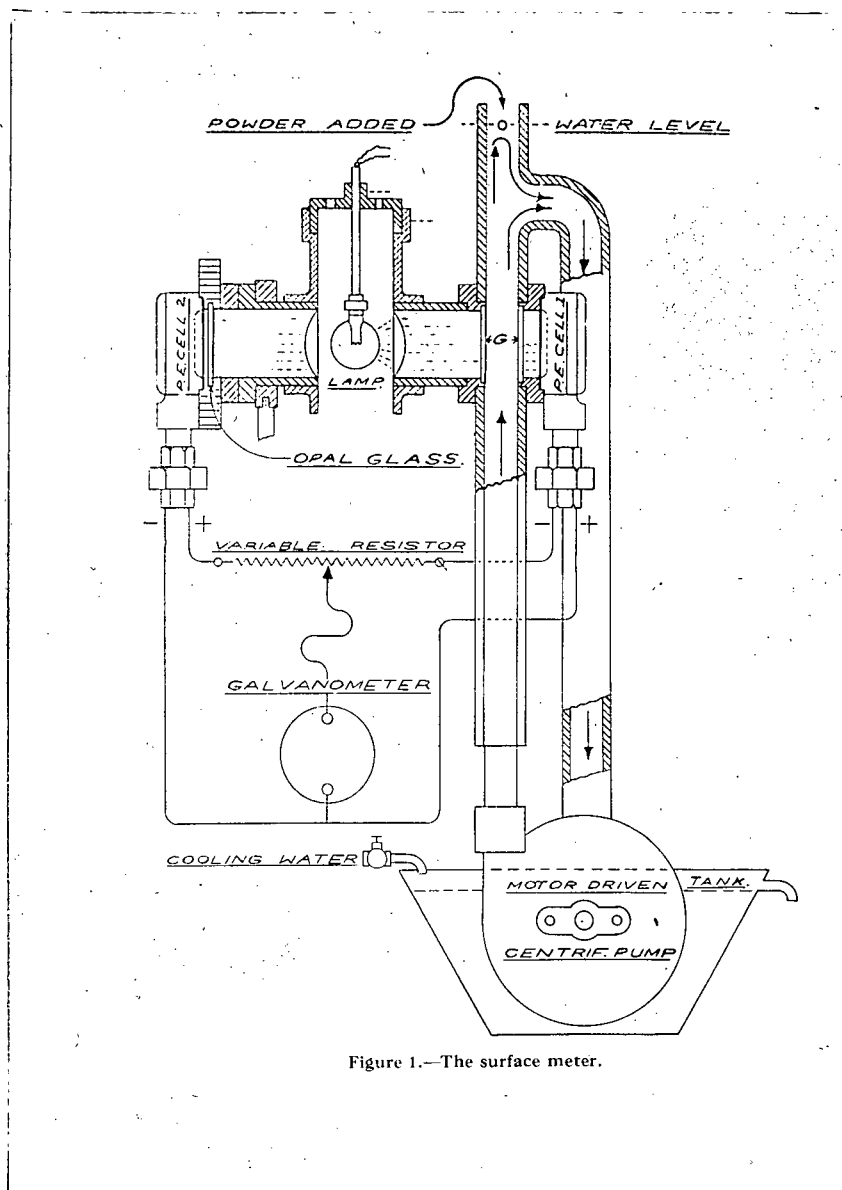


Figure 1.—The surface meter.

SCHEMATIC DIAGRAM OF BELL'S APPARATUS

Figure 1

for quartz $n_1 = 1.547$ (mean for D line)

water $n_0 = 1.335$

$R = .0054$

i.e. 0.54% of the incident light is reflected from a quartz water interface.

A graph showing the relation between refractive indices and the reflection coefficients of some materials used as white pigments in the paint industry is given in Figure 2.⁴⁾ It may be noted that the order of magnitude of the reflection coefficient of the materials, selected for their comparatively high degree of reflectivity for use in paints, is small. Thus it may be assumed that most materials used in turbidity measurements will exhibit a low reflection coefficient.

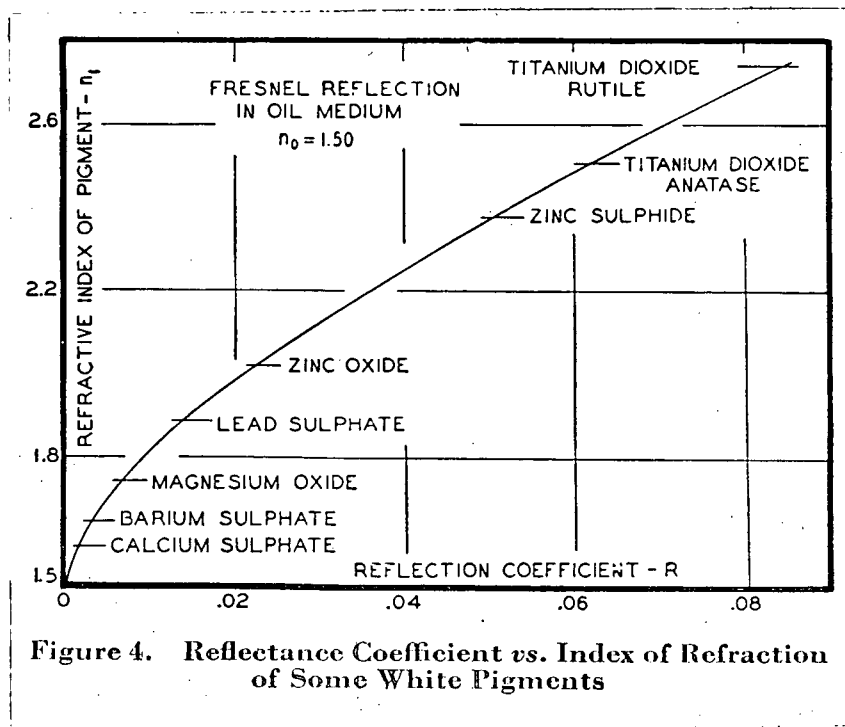


Figure 2

In the event that varying lustres and degrees of reflectivity of mixture of minerals might affect turbidity measurements the optical system of an apparatus such as Bell's should be designed so that suitable filters might be introduced. The use of light of a wave length far removed from that enjoying maximum reflection from mineral surfaces should make consistent results possible. This latter principle has been put to good use in the Fisher Electrophotometer which analyzes solutions containing dissolved substances that produce varying color intensities according to the amount dissolved.

There are a number of reasons why no great error should be expected due to polarization of the light passing through the fine particle suspensions. Insofar as transmitted light is concerned, all the light passing a polarizing medium should be absorbed except that part vibrating in a certain plane; consequently, as the light passes successively through particles with different orientations, it seems probable that only a small fraction of the initial light will remain unabsorbed.

In order to illustrate that the type of results expected from an apparatus such as Bell's could be predicted in advance the following theory may be hypothesized.

If a number of particles, considered as spheres of uniform diameter, are placed at random in the path of a beam of light and if it is assumed that the surface of the material is such that it will absorb 100% of the light which strikes it, an expression can be formulated which will give the change in light intensity as a function of the diameter of the particles, the specific gravity of the material and the weight involved.

~

Let I_0 = initial light intensity

I = final light intensity

therefore,

$$\frac{I_0}{I} = \frac{1}{1 - \% \text{ light cut off}}$$

where the % of light cut off

$$= \frac{\text{sectional area of particles}}{\text{sectional area of light beam}}$$

Let O_A = sectional area of particles

X = sectional area of light beam

W = weight of material

P = specific gravity of material

d_s = average particle diameter

N = number of particles

S = surface area of particles

$$= \pi d_s^2 N$$

$$O_A = \frac{\pi d_s^2}{4} N = 1/4 S$$

$$N = \frac{W/P}{\frac{1}{6} \times 3 \pi d_s^3}$$

$$O_A = \frac{W}{\frac{1}{6} \times 3 P \pi d_s^3} \frac{(\pi d_s^2)}{(4)} = \frac{3}{2 N P} W/d_s$$

Equation 2

$$\frac{I_0}{I} = \frac{1}{1 - \frac{3 \times 10^6 P (W/d_s)}{2 X}}$$

Equation 3

In the apparatus, however, the sectional area of the particles in the suspension as they pass in front of the photoelectric cells will always be a certain predetermined amount. Therefore O_A is constant and is equivalent to the amount of light cut off. It follows that,

$$d_s = \frac{3}{2 \pi P O_A} W = K W/P \quad \text{Equation 4}$$

K = a constant

which is the equation of a straight line passing through the origin. It must be remembered that the d_s referred to above is a surface mean diameter since it is actually determined from the expression,

$$d_s = \sqrt{\frac{\sum n d^2}{\sum n}} \quad \text{Equation 5}$$

It should be noted that if by a suitable arrangement of filters in the optical system of the apparatus it was found possible to obtain the same value for K (the slope of the calibration line) independently for different minerals it should be possible to measure, directly, the average surface diameters of mixtures of the different minerals.

III - THE APPARATUS

An apparatus, in many respects similar to Bell's device, was constructed. The apparatus consisted, essentially, of two phototubes, an electronic circuit for balancing the phototubes, a circulating chamber and a light source. The light source was mounted in a tee-shaped tube and a condenser lens mounted in each arm of the tee. The focal lengths of the

lenses were such that the rays of light after passing through them were substantially parallel. A plastic block in which glass windows had been set was attached to the end of one arm of the tee. On the other side of the plastic block was mounted one of the phototubes. The circulating tubes passed vertically through the plastic block. On the end of the opposite arm of the tee was attached another plastic block. A slot was cut in this block so that a piece of ground glass screen could be inserted thus cutting off a portion of the light reaching a phototube mounted on the other side of the block.

An electric eye vacuum tube (6E5), arranged in a bridge circuit, served to balance the outputs of the phototubes.

The intensity of the standard lamp was controlled by an A. C. ammeter.

Pictures and schematic diagrams of the apparatus are given in the following pages.

The first important pre-requisite in design of the circulating chamber was that the outlines of the chamber be extremely smooth such that there would be no sharp breaks in the direction of motion of the water, otherwise, local eddy currents would be set up and settling would occur. Secondly, the condition of turbulence must be such that the film of media on internal surfaces having zero velocity be of a minimum thickness, otherwise, the very fine portion of a mineral powder would have a tendency to adhere to the surfaces of the circulating chamber and be removed from circulation.

In accordance with these requirements the circulating chamber was built of clear plastic so that the motion of the fine particles could be observed. An impeller was installed which had sufficient power to prevent even the heaviest of minerals from settling out. The impeller shaft bearing

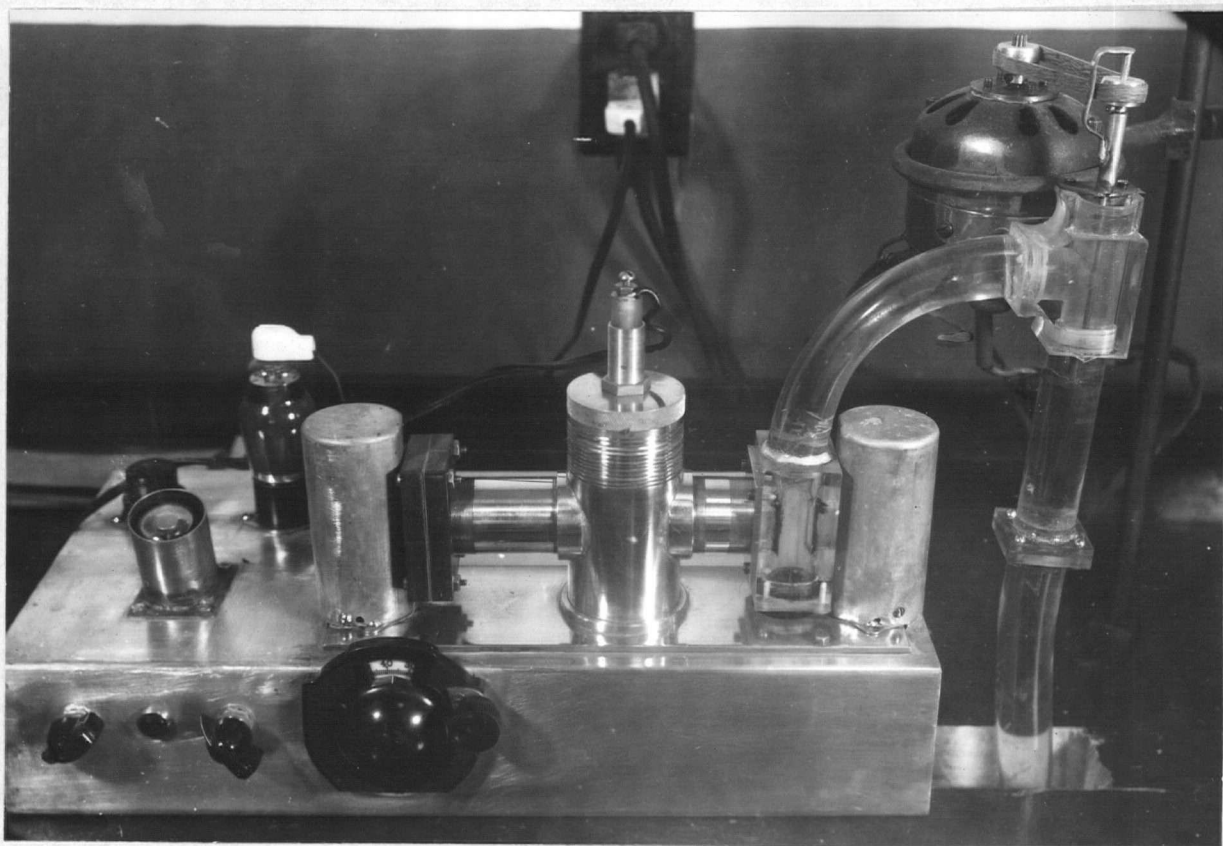


Figure 3

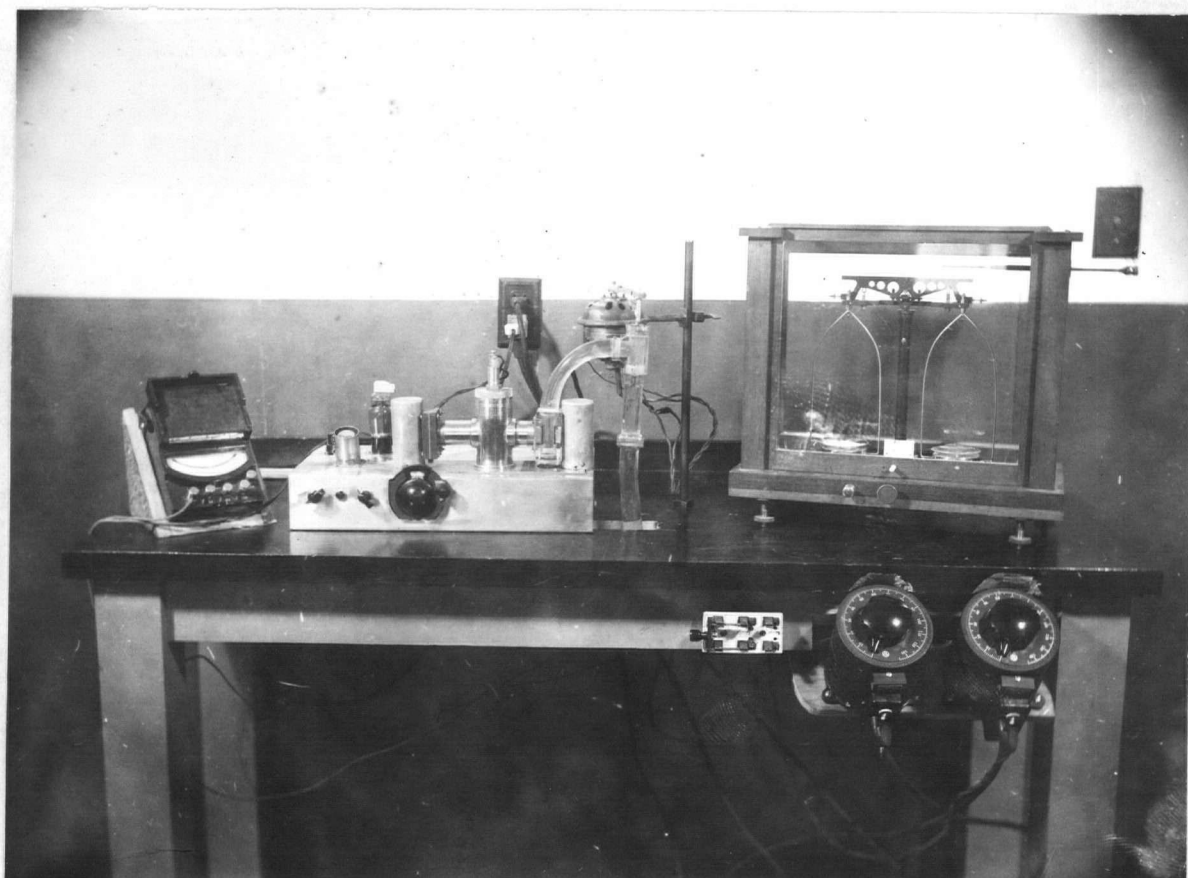


Figure 4

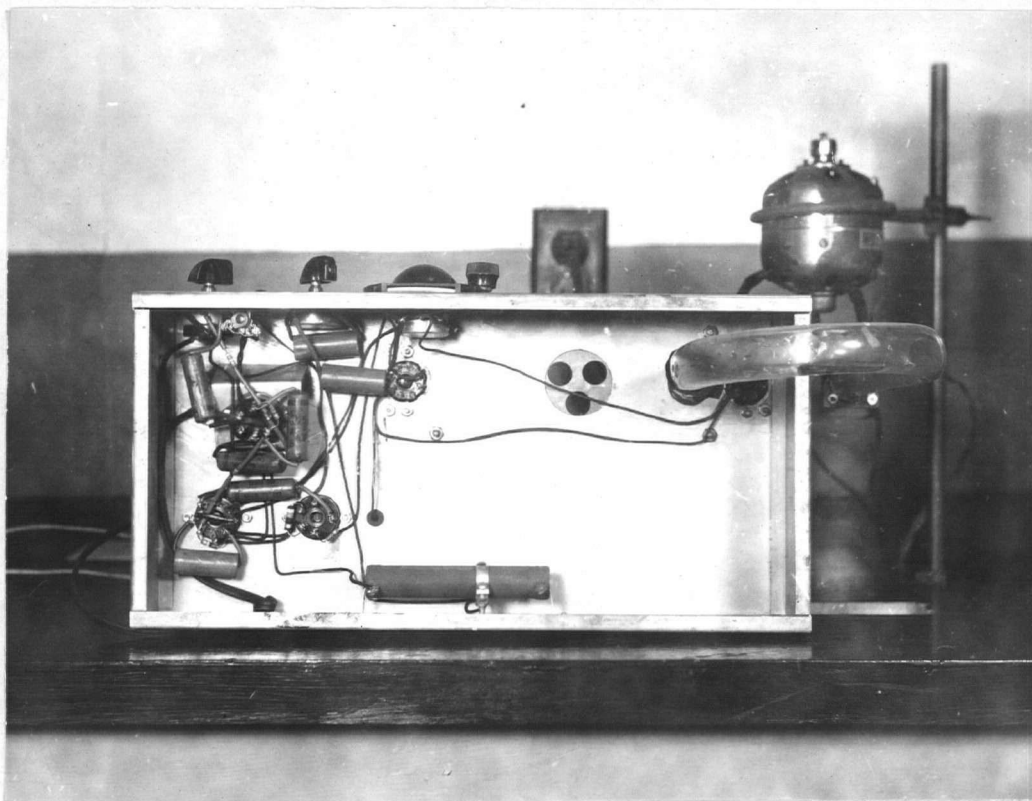


Figure 5

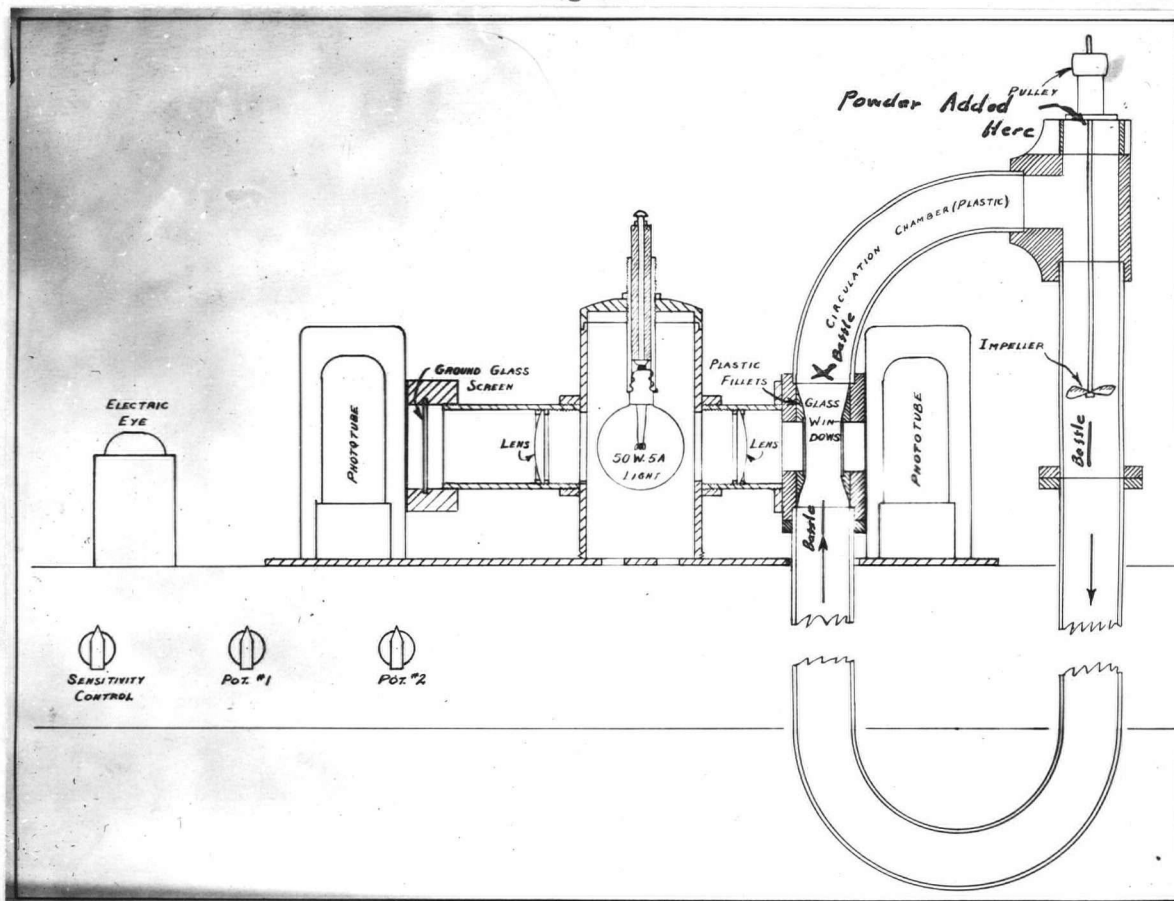


Figure 6

was mounted outside the circulating chamber in order to keep it from being damaged by abrasive materials and to keep the chamber free from any lubricating materials.

In the first few trial runs it was found necessary to install baffles of varied shapes in order to counteract a pulsating effect set up by the impeller. The position of the baffles is given in Figure 6.

The circulating chamber could possibly be further improved for some trouble has been experienced with extremely fine material adhering to the walls of the plastic tubes, especially at the bends. This fault is probably due to the surface of the lucite plastic. Although it would have been more difficult to construct the tubes of glass they would have been more satisfactory. However, if the plastic tubes are frequently cleaned with soap solution, consistent results can be obtained.

The electronic balancing circuit used in the apparatus has proven highly satisfactory. Once balanced, even moderately rough handling of the machine will not cause any noticeable drift or unbalance. The circuit employs two 929 phototubes and a 38 vacuum tube connected in the high sensitivity arrangement shown in Figure 7, but requires neither a battery nor a microammeter. According to the R.C.A. Manual from which this circuit was taken, $\frac{1}{4}$ of 1% unbalance in the light on the phototubes, at illumination levels as low as .0001 lumen, will cause the shadow angle on the 6E5 tube to open to 90° or close to 0° . At higher illumination levels, the 6E5 shadow angle gives full response to an even smaller percentage unbalance because the ratio of photoelectric current to leakage current is larger. Figures 8 and 9 give the spectral sensitivity curve and the anode characteristic curves of the type 929 phototube; the spectral sensitivity curve shows that this phototube is more sensitive to blue-rich light than to red-rich

light. The anode characteristic curves show that the response from the 929 phototube is a linear function of light intensity.

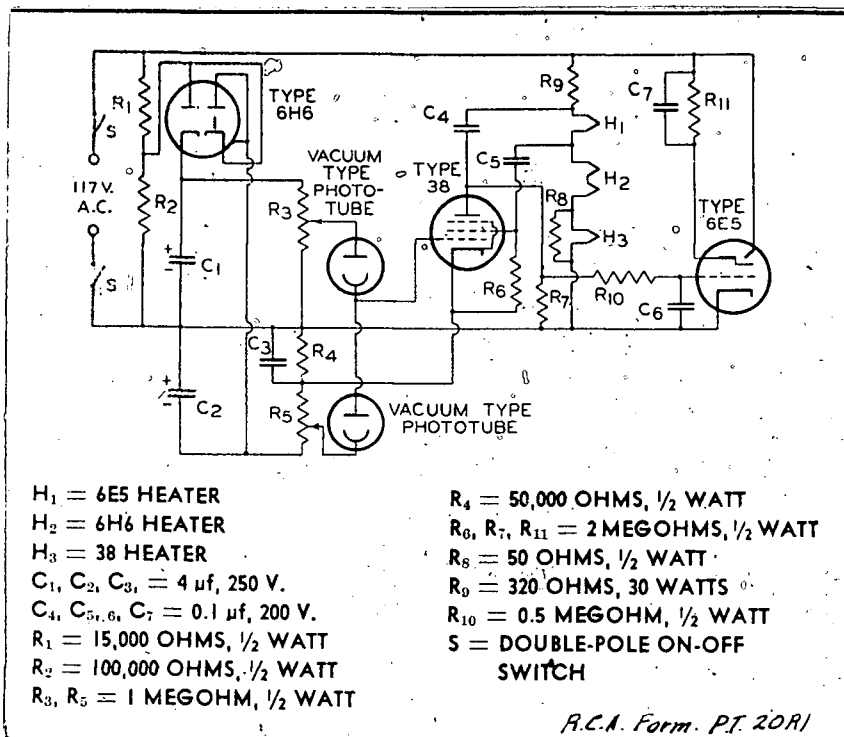


Fig. 9—A-C operated sensitive circuit for matching measurements.

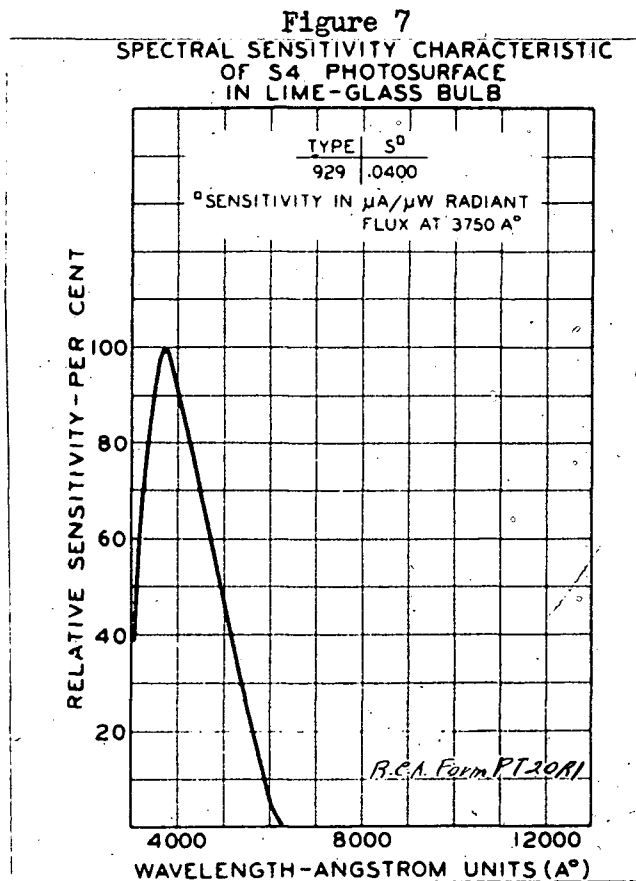


Figure 8

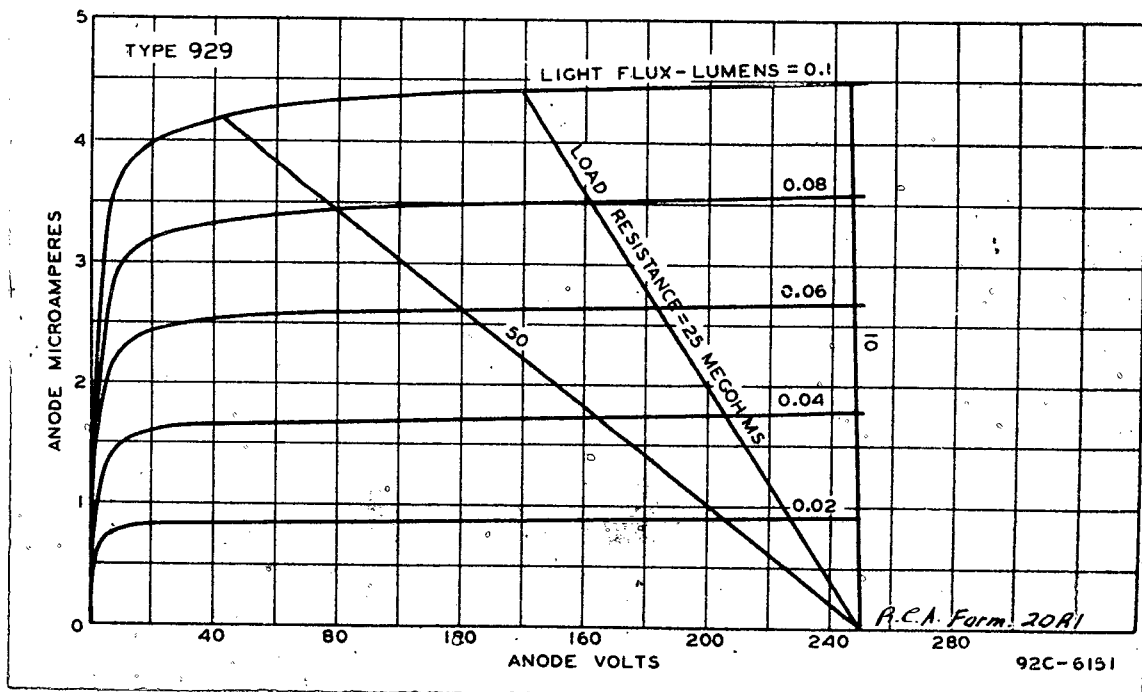


Figure 9

The procedure for determining surface area or particle size is as follows.

The circulation chamber is first filled to a certain mark with distilled water which has been previously boiled to remove as much of the dissolved gasses as possible. Air removal is necessary since gas bubbles have a tendency to form under the action of the impeller.

The impeller is started and the light source brought up to a certain intensity, determined by the A. C. ammeter.

The machine is then brought to balance by adjusting the two phototube potentiometers until the electric eye is just closed. The potentiometer shown on the right in Figure 3 is used for coarse adjustment

and the potentiometer on the left for fine adjustment. Once the initial balance is obtained it may be offset a fixed amount by inserting the ground glass screen in the slot.

The machine is now brought to balance once more by adding to the circulation chamber the material on which the determination is to be made. As given in Equation 3 the weight necessary for this final balance will be a direct function of the average surface area diameter of the material.

IV - PREPARATION OF SAMPLES FOR CALIBRATION

It was evident at the beginning of the work on fine particle measurement that success or failure would depend primarily upon the standard powders used in the calibration of the apparatus. Therefore it was of great importance that the average particle size of these powders be determined accurately.

The most desirable method of particle size determination would be applicable to a number of minerals without regard to chemical composition and would also give some measure of particle shape. Further, the use of mineral powders, that had been sized and graded, would obviate the necessity of determining complicated size distribution curves.

The Haultain Infrsizer was used for the preparation of calibration samples since it will separate a -325 Mesh mineral powder into six uniformly graded groups. The six groups will have average particle sizes ranging from 10 microns to 50 microns.

About 200 grams of material was charged to the Infrsizer for a single run. The time required for treatment was approximately one hour for every 100 grams of charge. The runs, for the samples to be used in

calibration, were made at air pressures varying from 24" H₂O to 28" H₂O.

Since the Infrsizer is, in effect, an air elutriation device for the grading of fine material, the shape of the individual particles will be an important factor in efficiency of grading. Photographs of glass spheres ⁷⁾, that had been graded by the Infrsizer, showed that the degrees of sorting, according to size, is particularly good. In the accompanying pictures of mineral powders, (quartz, chalcoppyrite, pyrite), where the shape of the particles is not uniform, the closeness of sizing is still remarkably good. The standard deviations and standard errors, given in Table I indicate the efficiency of the Infrsizer.

One or two photomicrographs of samples from each sized group of mineral powders were taken on 5" x 7" plates at magnifications varying from 80 to 400 times.

Under these conditions from 150 to 250 particles were included in the area photographed. A few milligrams of powder, taken from a group, were placed on a clean glass slide. The addition of a drop of Xylene accompanied by slight side tapping of the slide served to spread the powder into a thin layer. The Xylene, when dry, also provided the adhesion necessary to hold the particles on the slide. The photomicrographs were taken on a Leitz Metallograph using dark field illumination in the case of quartz and direct illumination in the case of pyrite and chalcoppyrite.

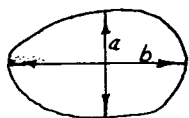
An eyepiece with a graduated scale was used in the microscope in order that a reference scale would appear on the photomicrographs. This scale was calibrated, with respect to the various objectives used, by comparing it with a standard 1 mm scale mounted on the microscope specimen table.

The photomicrographs were projected on a 4 foot by 5 foot screen and the value of each projected scale division determined in inches. A

standard inch ruler, graduated in tenths, was used to measure the diameters of the projected images.

Two hundred particles were considered a representative sample of each size group. Two length dimensions of each particle were taken. These measurements, which have been termed the critical particle dimensions, correspond to the longest and shortest axes of the particle. The square root of the product of these individual measurements was taken as the surface diameter of the particle under consideration. The reasons underlying this procedure were, firstly, that the surface diameter must necessarily be determined from some dimension to the second power denoting area and, secondly, that with one mineral there is assumed to be a constant shape factor which, if multiplied by the product of two particle dimensions i.e. the surface diameter squared, will give the sectional area of the particle. Previously (Page 14), it has been shown that the sectional area of particles in a suspension is a function of the total area of the particles and that it is the sectional area that will be measured by the photometric apparatus. The particle shape factor will have some value between 1, for a rectangle, and $\frac{1}{4}$, for a circle.

The products of the short and long dimensions of all the particles measured were added and this divided by the number of particles measured. The square root of the resultant figure was taken as the average particle diameter for the sized group under consideration. The above procedure would be mathematically expressed as follows.



a, b = dimensions measured through the centroid of the particle

$$d_s(\text{average}) = \sqrt{\frac{\sum (ab)}{N}}$$

$d_{s(av)}$ = average surface diameter

N = number of particles

The root mean square method of average particle size calculation thus weights the surface area presented by the larger particles and consequently the average diameter arrived at in this manner is a better measure of the surface area of a group of particles than the arithmetic average diameter.

The average surface diameters determined for the sized groups of the minerals, (quartz, chalcopyrite, and pyrite), are given in Table 1. Standard deviations and standard errors of these average surface diameters are also given. The formula used for the calculation of the standard deviation is as follows;

$$\sigma = \sqrt{\frac{\sum (d_s - d_{s(av)})^2}{N}}$$

σ = standard deviation

N = number of particles

and that used for the calculation of the standard error is as follows;

$$\hat{\sigma} = \frac{\sigma}{\sqrt{N}}$$

$\hat{\sigma}$ = standard error

Figure 10 is a frequency distribution graph of the six groups of powders obtained from the Haultain Infrsizer. For the sake of simplicity, the maximum ordinates of all the groups have been made equal. It will be noted that the grading of finer particles is closer than that of the larger particles.

Figures 12 to 16 and 18 to 22 are photomicrographs of samples from the six size groups obtained from the Infrsizer. Figures 17 to 23 are photomicrographs of samples from the Infrsizer dust bag overflow.

It was of interest to estimate the value of the shape factors for the minerals that were to be used in the surface area and particle diameter determinations. A frequency graph, showing the percentage deviation of the largest measurement taken for the diameter of a particle from the root mean square diameter of that particle, is given in Figure 11 for the mineral quartz. The peak of the frequency curve indicates that the longest dimension of the average quartz particle would be 35% longer than the surface area diameter of the particle. The shortest dimension of the particle would be 35% less than the surface area diameter of that same particle. If the average quartz particle cross-section was considered an ellipse, with the critical dimensions as outlined above, the value for the calculated shape factor would then be .895 .

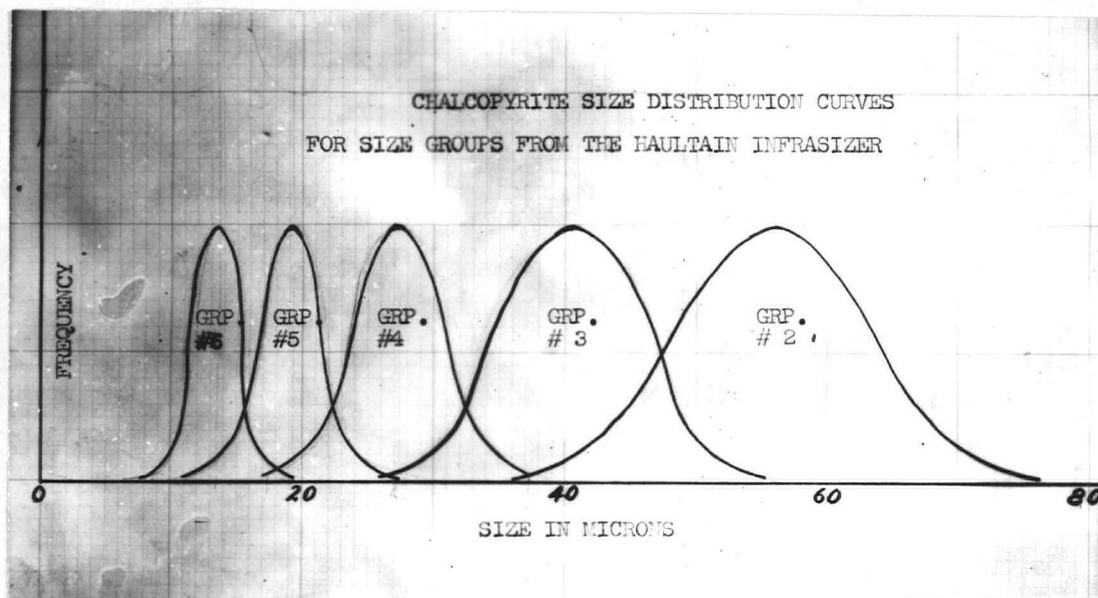


Figure 10

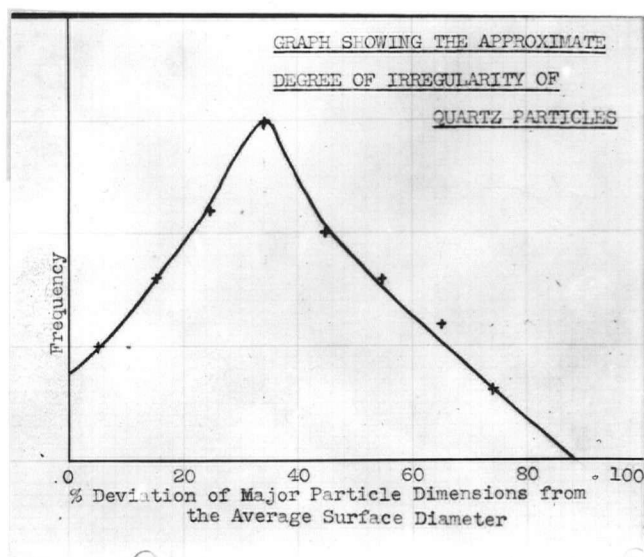


Figure 11

MATERIAL	GROUP NO.	AVERAGE SURFACE DIAMETER	σ , STANDARD DEVIATION	$\frac{\sigma}{\bar{x}}$, STANDARD ERROR
Quartz	6	18.7 μ	5.7 μ	0.61 μ
	5	28.6 "	7.7 "	0.80 "
	4	39.5 "	6.7 "	1.05 "
	3	48.5 "	8.2 "	1.03 "
	2	74.3 "	9.7 "	1.19 "
	7			
Chalco- pyrite	6	13.6 "	2.5 "	0.32 "
	5	19.4 "	4.1 "	0.40 "
	4	27.3 "	5.0 "	0.48 "
	3	40.5 "	6.8 "	0.87 "
	2	56.0 "	8.8 "	1.38 "
Pyrite	6-1	13.1 "	3.0 "	0.32 "
	5-1	18.2 "	3.1 "	0.29 "
	4-2	27.3 "	4.3 "	0.48 "
	3-2	39.0 "	6.4 "	0.58 "
	2-2	51.9 "	8.1 "	1.26 "

Table No. I

Figure 12

Quartz

Sample #2

156 X

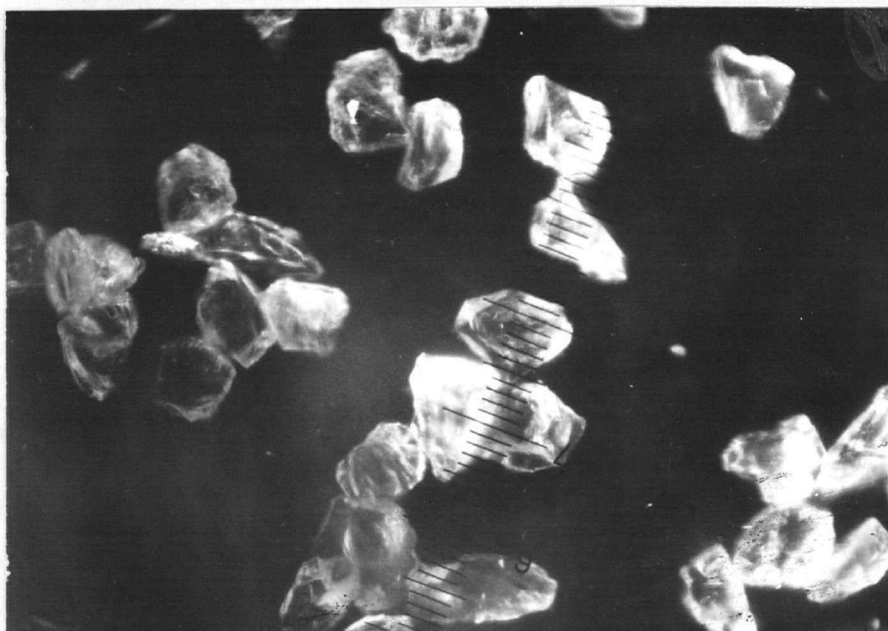


Figure 13

Quartz

Sample #3

156 X

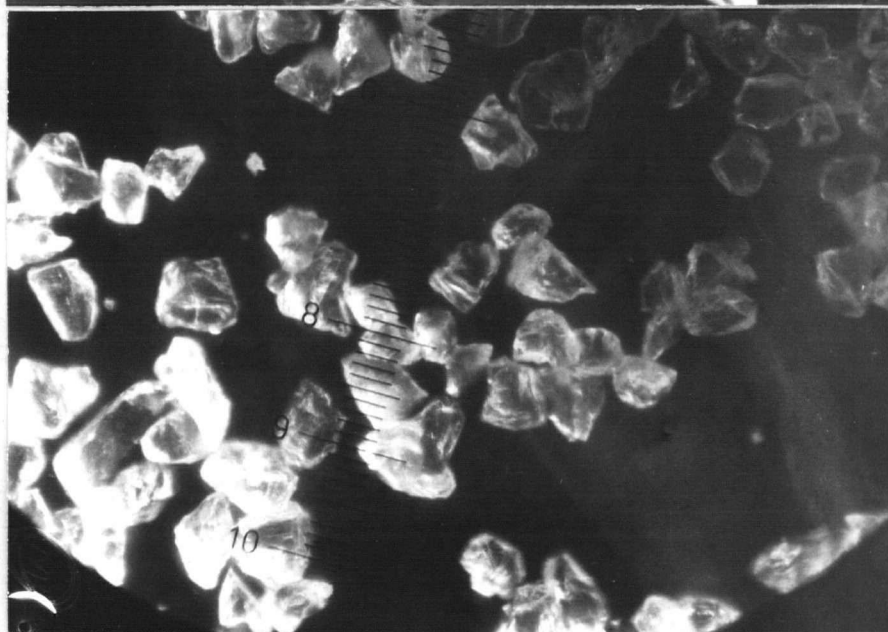


Figure 14

Quartz

Sample #4

174 X

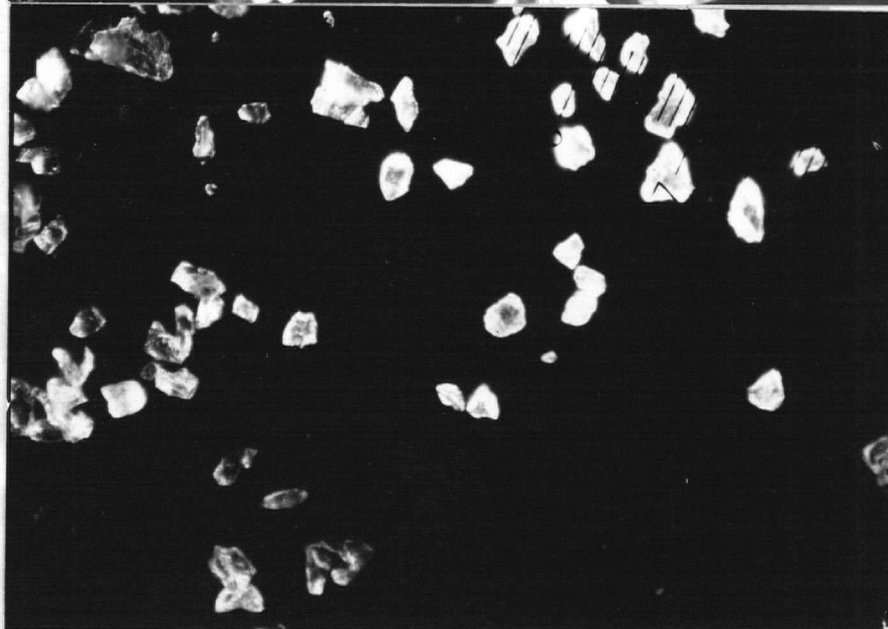


Figure 15

Quartz

Sample #5

126 X

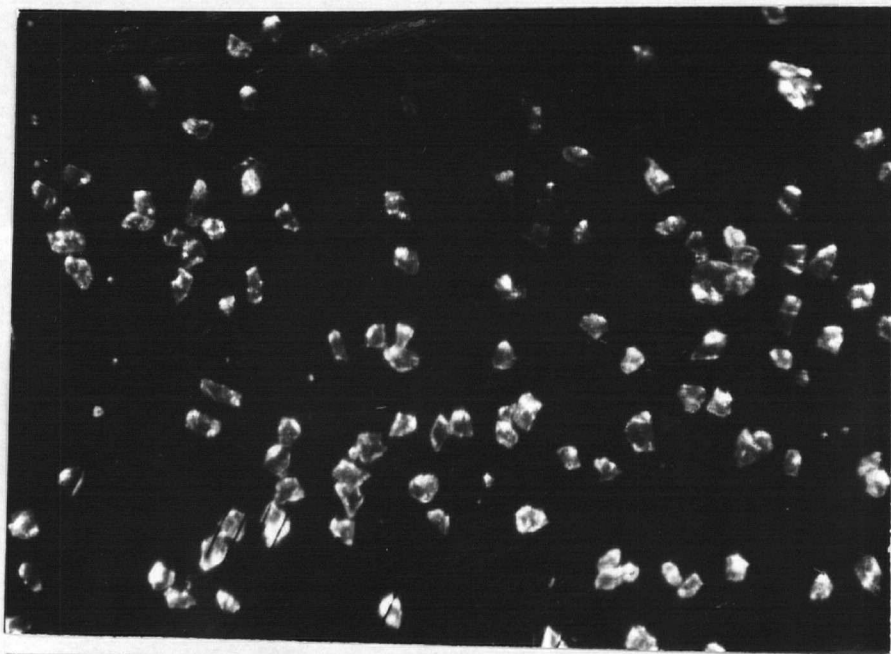


Figure 16

Quartz

Sample #6

408 X

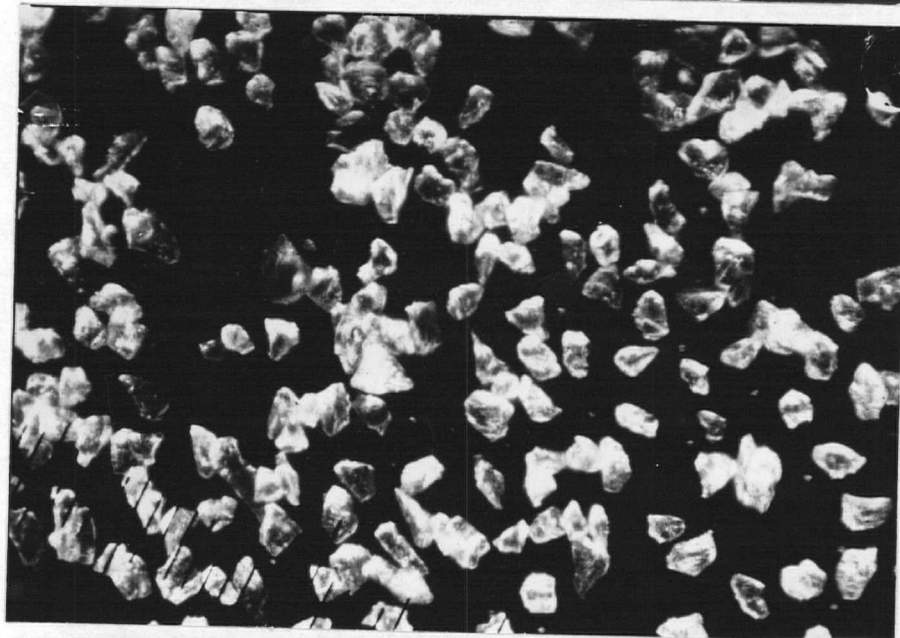


Figure 17

Quartz

Sample #7

247 X

*Infrasizer Dust
Bag Overflow*

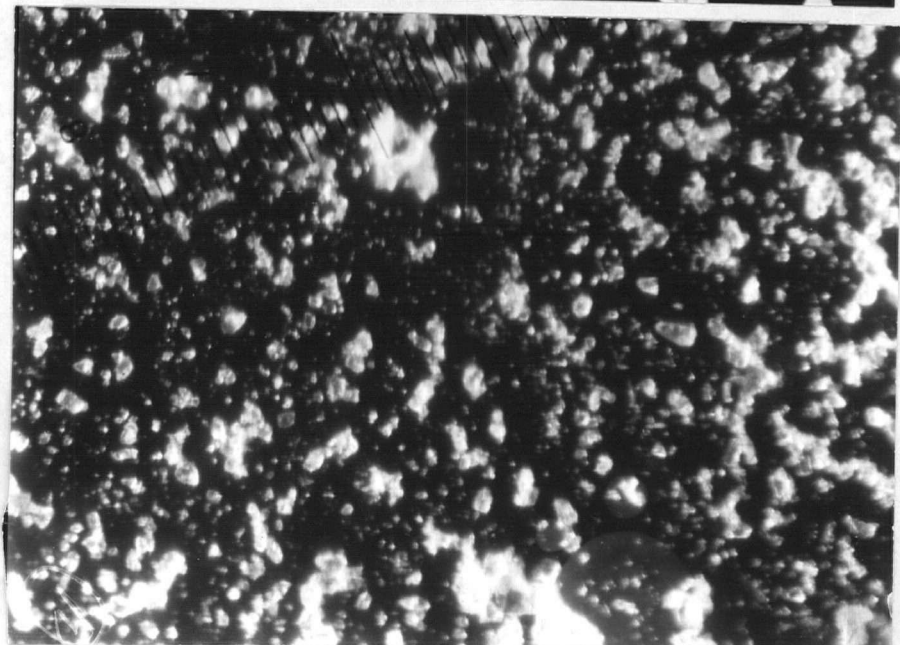


Figure 18

Pyrite

Sample # 2-2

82 X

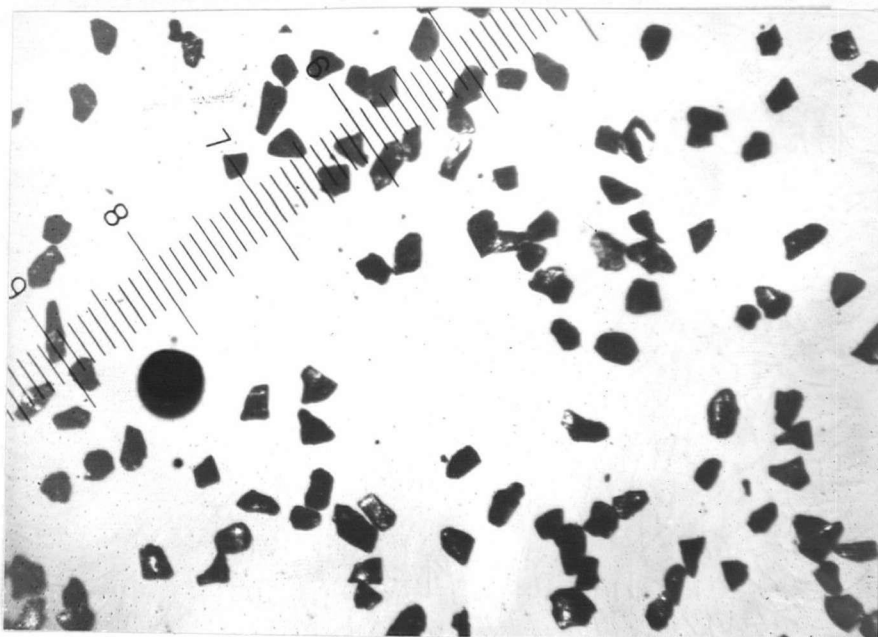


Figure 19

Pyrite

Sample # 3-2

90 X

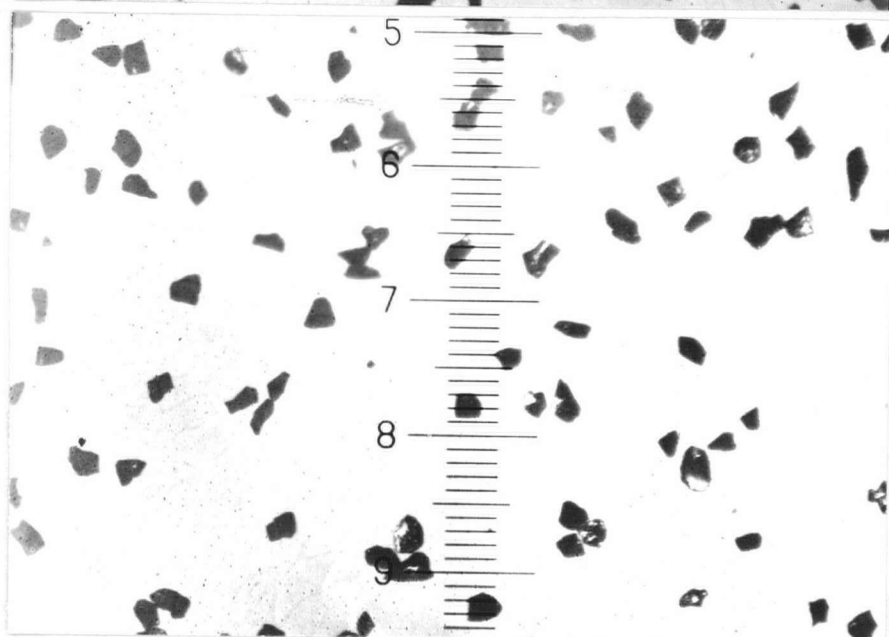


Figure 20

Pyrite

Sample 4-2

170 X

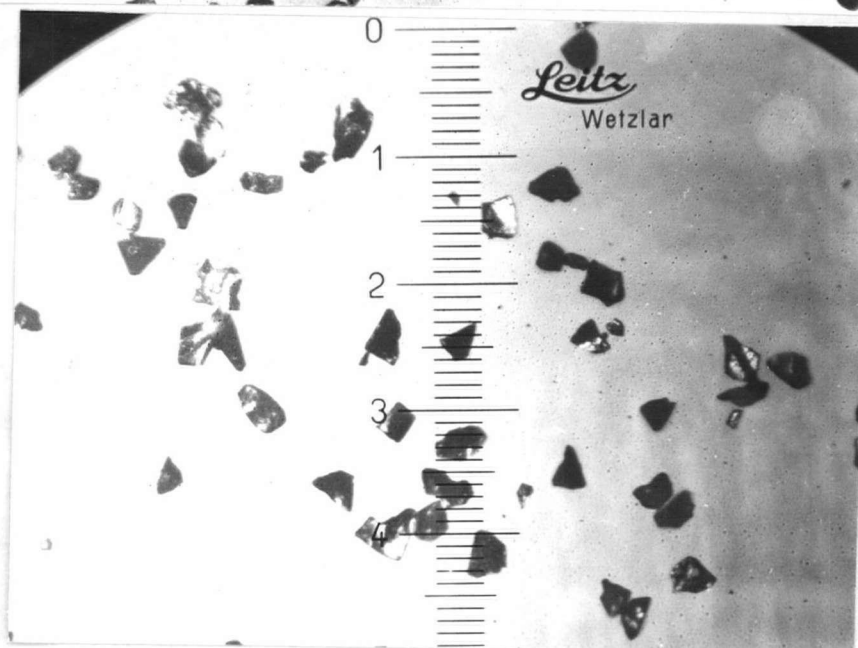


Figure 21

Pyrite

Sample 5-1

170 X

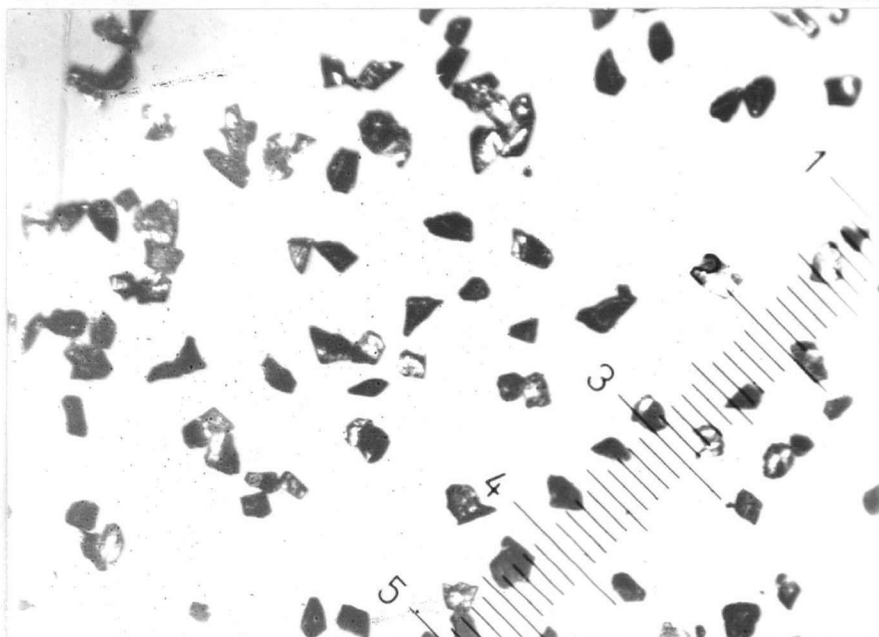


Figure 22

Pyrite

Sample 6-1

205 X

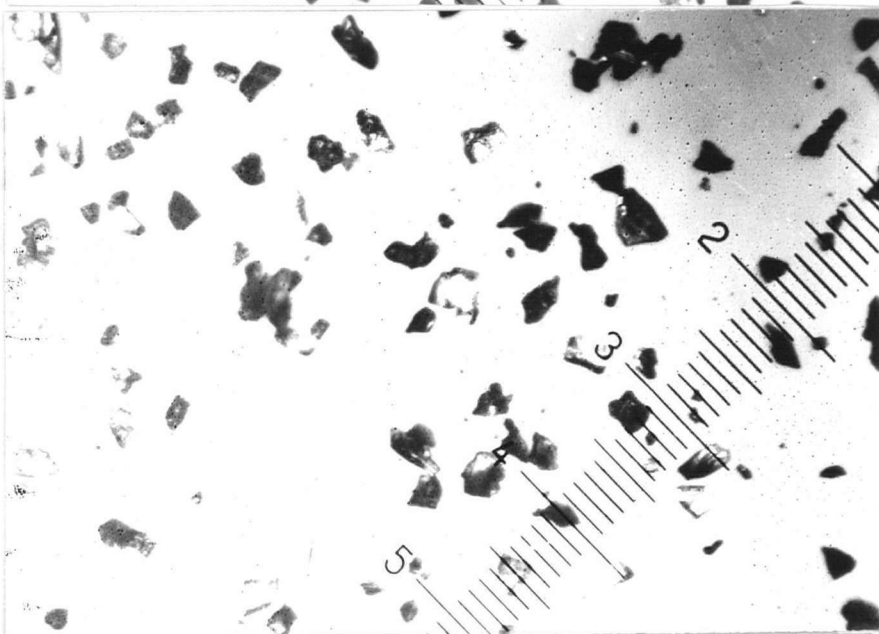


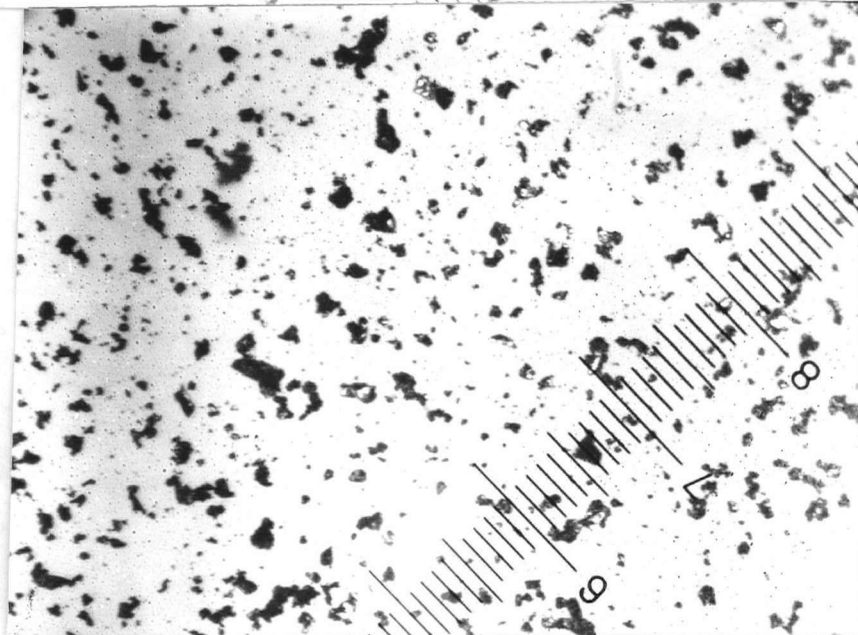
Figure 23

Pyrite

Sample 7-0

283 X

*Infrasizer Dust
Bag Overflow*



V - EXPERIMENTAL RESULTS

The sized groups of minerals which had been prepared in the Infralyzer machine and the weights-to-balance for each size group *were* determined. The results are given in Table II.

Calibration graphs were drawn up showing the weight-to-balance versus the average surface particle diameter as given in Table II. The calibration graphs for quartz, chalcopyrite, and pyrite are given in Figures 24, 25, and 26.

In order that some correlation of the calibration graphs for the three minerals used might be obtained the specific gravity of the individual minerals must be taken into account as given in formula 2. Figure 27 is a graph in which W/P is plotted against the average surface diameters for the size groups of the minerals.

It will be noted that all the calibration graphs show, as expected, a straight line relationship between particle size and the weight-to-balance. It is of particular interest to note that the calibration lines, for the three minerals investigated, pass through the origin. It may be concluded from this fact that the effects of polarization, reflectivity, refraction, birefringence etc., of the fine particles in the apparatus, are negligible.

Since quartz exhibits a fair degree of transparency and the minerals chalcopyrite and pyrite are opaque, it was expected that the slope of the calibration line for quartz would be less than those for chalcopyrite and pyrite and such has proven to be the case.

In the final calibration graph, Figure 27, it is seen that the calibration lines for pyrite and chalcopyrite coincide. This fact

MATERIAL	GROUP NO.	AVERAGE SURFACE DIAMETER	W, WEIGHT TO BALANCE	W/P
Quartz	6	18.7 μ	1.358 gms.	.502
P = 2.7	5	28.6 "	2.090 "	.774
	4	39.5 "	3.077 "	1.139
	3	50.2 "	3.930 "	1.455
	2	73.7 "	5.685 "	2.102
	7			
Chalco- pyrite	6	13.6 "	.748 "	.182
P = 4.1	5	19.4 "	1.105 "	.270
	4	27.3 "	1.345 "	.328
	3	40.5 "	2.090 "	.510
	2	56.0 "		
Pyrite	6-1	13.1 "	.729 "	.146
P = 5.0	5-1	18.2 "	1.090 "	.218
	4-2	27.3 "	1.721 "	.344
	3-2	39.0 "	2.540 "	.508
	2-2	51.9 "	3.419 "	.685

Table No. II

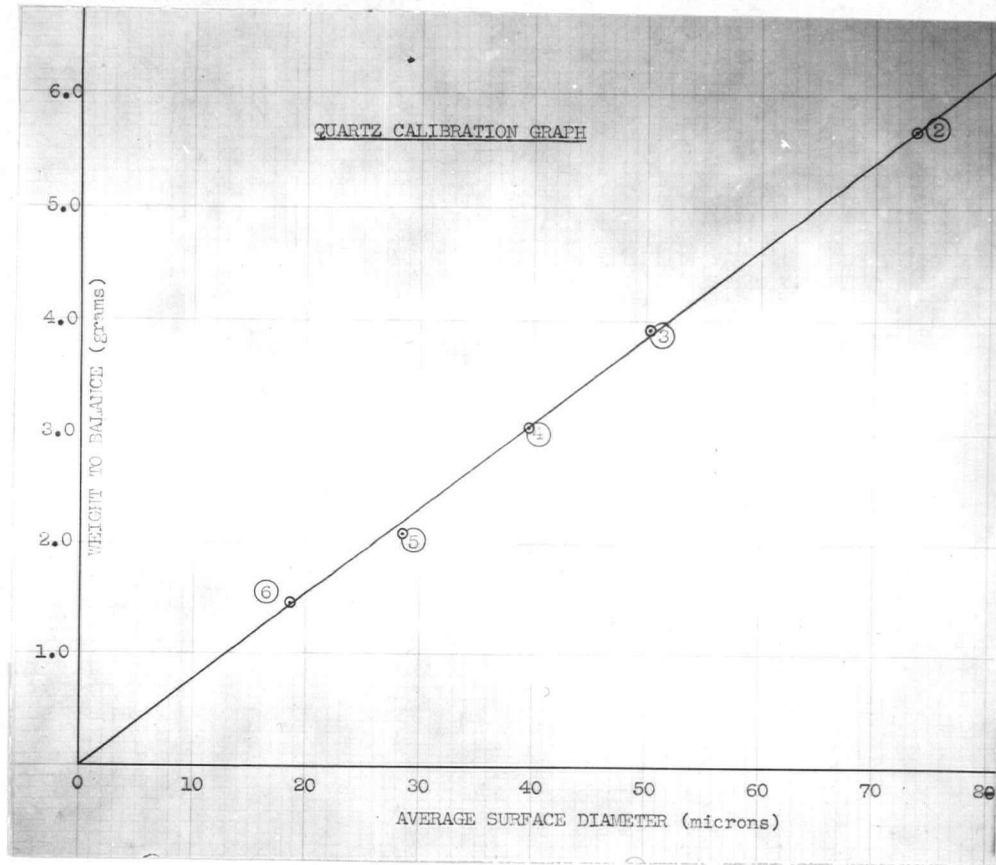


Figure 24

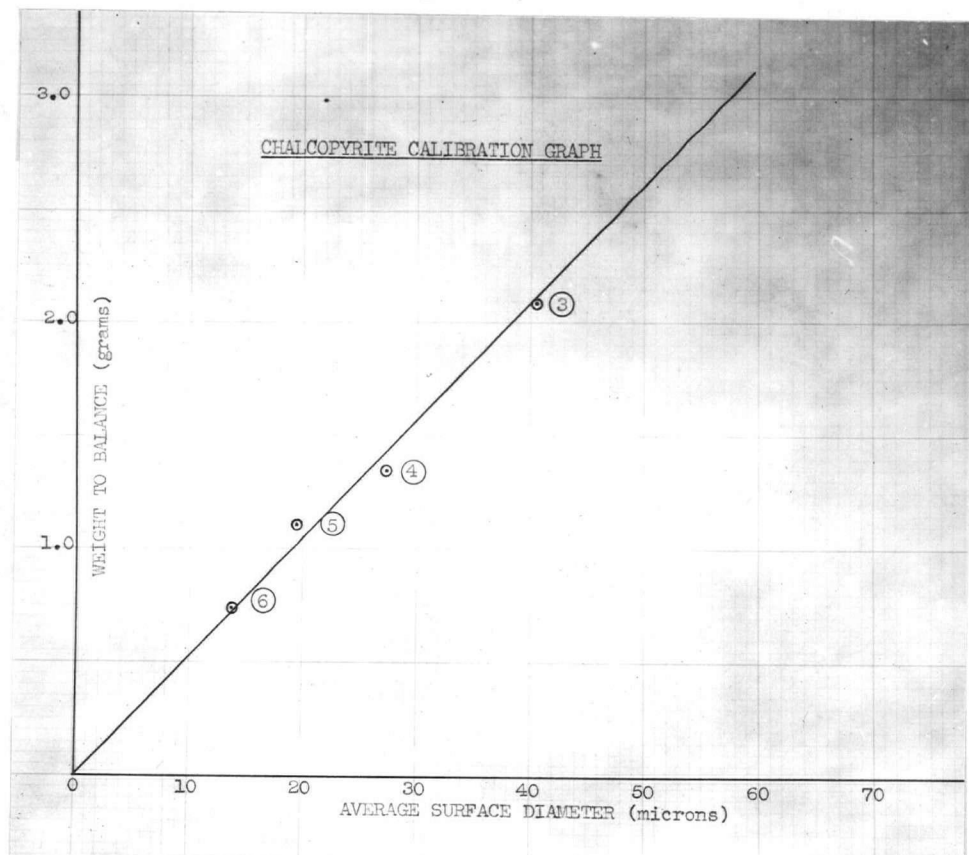


Figure 25

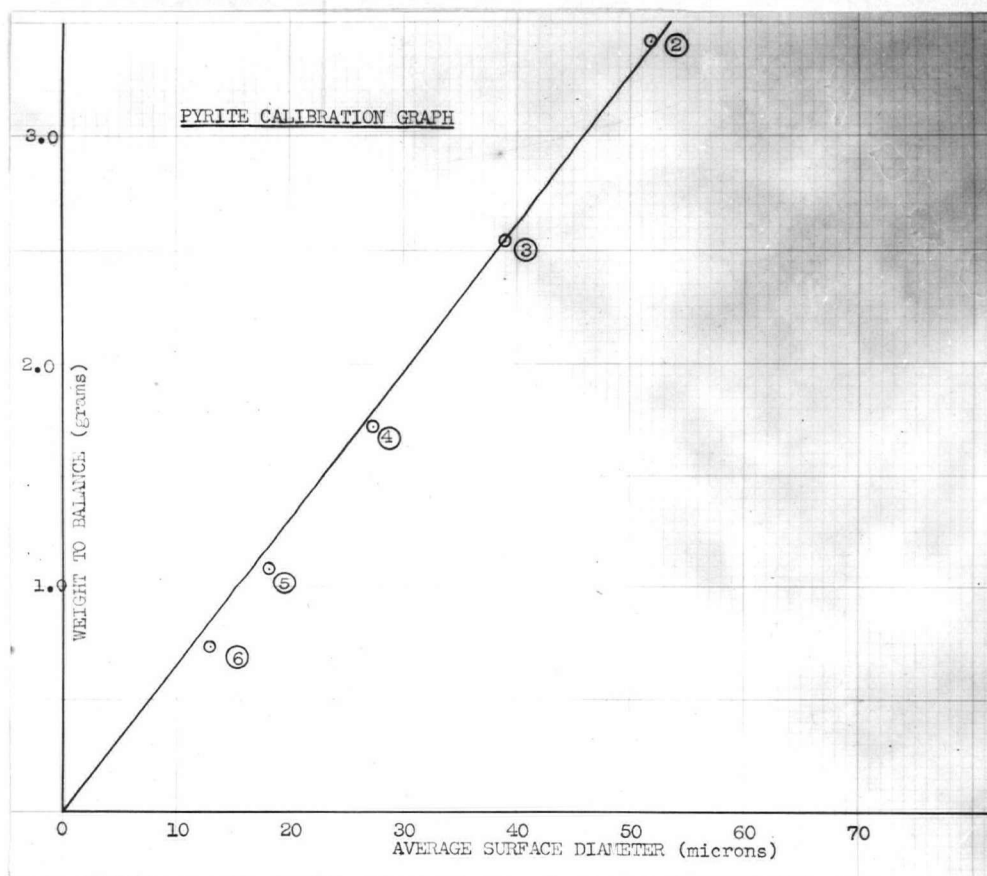


Figure 26

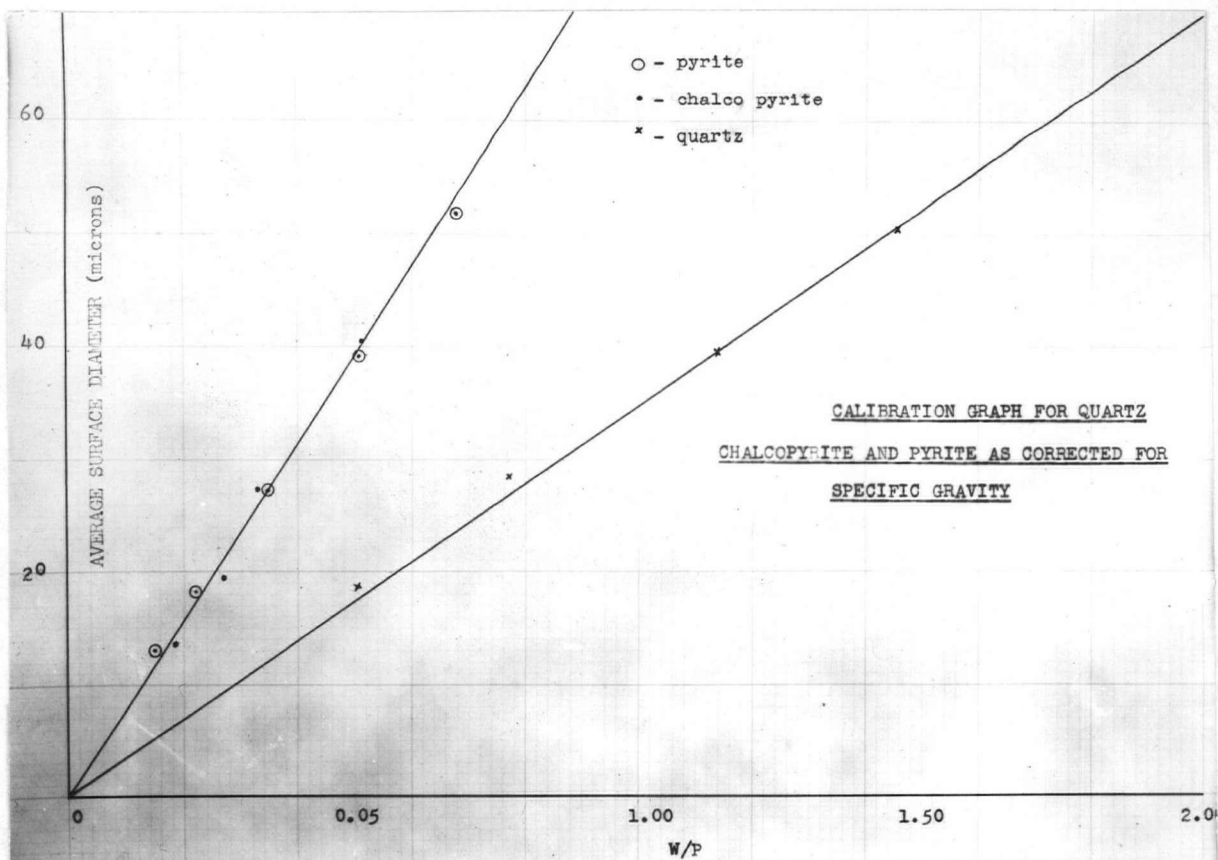


Figure 27

suggests that any opaque material, when corrected for specific gravity, would have the same calibration line and that surface areas and average diameters of mixtures of opaque materials may be directly determined.

A few tests were made on mixtures of different size groups of the same mineral to determine whether the apparatus would satisfactorily measure the average surface diameters of a material where the range of particle size was large. Following are the results of a test.

Mat'l & Sample No.	Individual Wt.-to-Bal.	Composite Wt.-to-Bal.	Fraction of Bal.	Av.Part. Size	Av. Calc. Part. Size
quartz #6	1.358 gms.	.800 gms	.590	18.7 M	31.5 μ
quartz #3	3.930	$\frac{1.465}{2.265}$ "	$\frac{.373}{.963}$	50.2	
<div style="text-align: right;"> % Error of Bal. = 3.7% </div>					

Table No. III

The average surface particle size determined from the graph is 30.0 μ .

In order that the photometric method of fine particle measurement could be applied to mixtures of opaque and partially transparent minerals a graph as shown in Figure 28 was drawn up. This graph is the same as that of Figure 27 except that a series of lines representing the volume assay of quartz, chalcopyrite mixtures, has been drawn between the calibration lines for pure quartz and pure chalcopyrite (or pyrite).

The slopes of the lines in Figure 28 were determined in the following manner.

With reference to formula 2, it may be remembered that

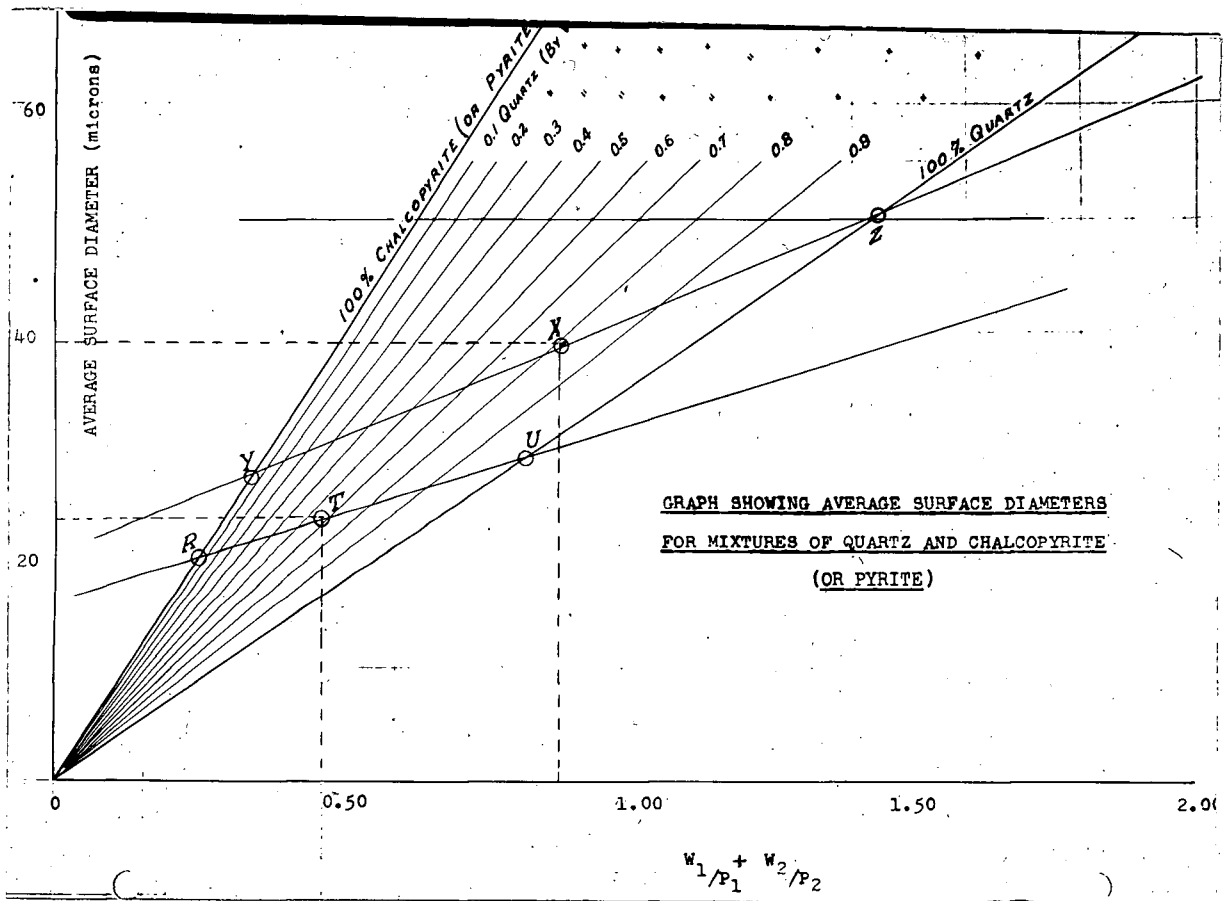


Figure 28

O_A is the specific surface necessary to bring the apparatus to balance. This surface, with mixtures, is made up of all the surfaces presented by the components of the mixture.

$$O_A \text{ (for 100\% quartz)} = \frac{W/P}{.5236 d^3} (\pi d^2/4)$$

$$= A (W/Pd)$$

Similarly, O_A (for 100% chalcopyrite)

$$= B (W/Pd)$$

The variance in the constants A and B will depend on the relative transparency of the materials under consideration, i.e. quartz and chalcopyrite.

Therefore with mixtures,

$$O_A = A \frac{W_1}{P_1 d_1} + B \frac{W_2}{P_2 d_2}$$

where,

A = a constant

B = a constant (not necessarily the same value as A)

W_1 = amount of quartz in the mixture used to balance
the apparatus

P_1 = specific gravity of quartz

W_2 = amount of chalcopryrite in the mixture used to
balance the apparatus

P_2 = specific gravity of chalcopryrite

d_1 = average surface diameter of the quartz particles

d_2 = average surface diameter of the chalcoprybite particles

and with specific reference to quartz and chalcopryrite,

$$B = 2.24 A$$

where B = equals slope of the chalcopryrite line

and A = equals slope of the quartz line.

$$K = \frac{W_1}{P_1 d_1} + 2.24 \frac{W_2}{P_2 d_2}$$

This is the equation of a straight line passing through the
points $(W_1/P_1, d_1)$ and $(W_2/P_2, d_2)$ on the graph.

For simplicity a horizontal line was selected.

$$\text{i.e. } d_1 = d_2 = 50 \mu$$

and

$$K_1 = W_1/P_1 + 2.24 W_2/P_2$$

$$\text{If } W_1/P_1 = 0$$

$$K_1 = 1.41$$

$$\therefore 1.41 = W_1/P_1 + 2.24 W_2/P_2$$

The ratio $\frac{W_1/P_1}{W_2/P_2}$ was given the values 9/1, 8/2, 7/3, 6/4, 5/5, 4/6, 3/7, 1/9, and the values for $W_1/P_1 + W_2/P_2$ determined as follows.

$W_1/P_1 / W_2/P_2$	W_1/P_1	W_2/P_2	$W_1/P_1 + W_2/P_2$
9/1	1.130	.125	1.225
8/2	.904	.226	1.130
7/3	.718	.308	1.026
6/4	.565	.377	.942
5/5	.435	.435	.870
4/6	.323	.485	.808
3/7	.226	.528	.754
2/8	.141	.566	.707
1/9	.066	.600	.666

Table No. IV

The values for $W_1/P_1 + W_2/P_2$ were plotted along the 50μ line and straight lines drawn from each point through the origin. As mentioned before these lines represent the volume assay of the mixture.

A few runs of chalcopryite-quartz mixtures were made to determine the reliability of the calculated graph.

Mat'l	Size	W	Comp.Wt.- to-Bal.	W/P	% O _A	% Vol.
Quartz #3	50.2	3.930g	2.000 g	.740	50.9	83.3
Chalco. #4	27.3	1.345 "	.638 "	.155	47.5	16.7
				<u>.895</u>	<u>98.4</u>	<u>100.0</u>
					% Error of Bal. = 2%	

Table No.V

Average surface diameter

$$= \sqrt{\frac{(.740/50.2 + .155/27.3)}{\frac{.740}{(50.2)^3} + \frac{.155}{(27.3)^3}}} = 39.4 \mu$$

(point X on the graph)

The value of the average diameter from the graph is approximately 39μ .

Mat'l	Size	W	Comp.Wt.- to-Bal.	W/P	% O _A	% Vol.
quartz #5	28.6	1.930g	.900 g	.333	46.5	72.0
Chalco. #5	19.4	1.105 "	.580 "	.130	53.4	28.0
				<u>.463</u>	<u>99.9</u>	<u>100.0</u>

Table No. VI

Average surface diameter

$$= \sqrt{\frac{(.333/28.6 + .130/19.4)}{\frac{.333}{(28.6)^3} + \frac{.130}{(19.4)^3}}}$$

$$= 24.0 \mu . \quad (\text{point T on the graph})$$

The value of the average diameter from the graph is approximately 23.6μ .

In summary the procedure in using the graph of Figure 28 would be as follows.

Firstly, determine the weight assay of the material to be used and convert to a volume assay. Secondly, determine the weight-to-balance for the mixture and convert to the volume-to-balance i.e.

$$W_1/P_1 + W_2/P_2$$

Lastly, read off the average surface diameter corresponding to the value for the volume-to-balance from the correct volume assay line on the graph.

In Figure 28, the lines YXZ and RTU are of no particular interest except that they provide a secondary check on the validity of the graph. The following might be mentioned, however, as a practical use of such lines. If it was of interest to determine the differential rate of size reduction of the sulphide fraction of an ore as compared to that of the gangue fraction then a line such as YXZ or RTU would furnish this information. A test on the ground

product of the ore would determine one point on the graph. Then if it was possible to disturb the volume assay of this ground product, say by panning, another test could be made and another point plotted on the graph. The intersections of the line joining these two points with the 100% sulphide line and the 100% gangue line would then give the average diameters of the sulphide and the gangue fractions of the ground product.

Figure 29 is a plot of specific surface area versus the reciprocal of the weight to balance. Since no absolute values of surface area were available for calibration of the apparatus, it has been necessary to introduce a constant, B , termed the irregularity factor, which when multiplied by the calculated specific surface area of a material, will give the absolute specific surface area of that material. B will usually have a value between 1.0 and 1.30. The introduction of such constants in particle size work is often necessary. However, they do not usually detract from the value of the data to which they pertain.

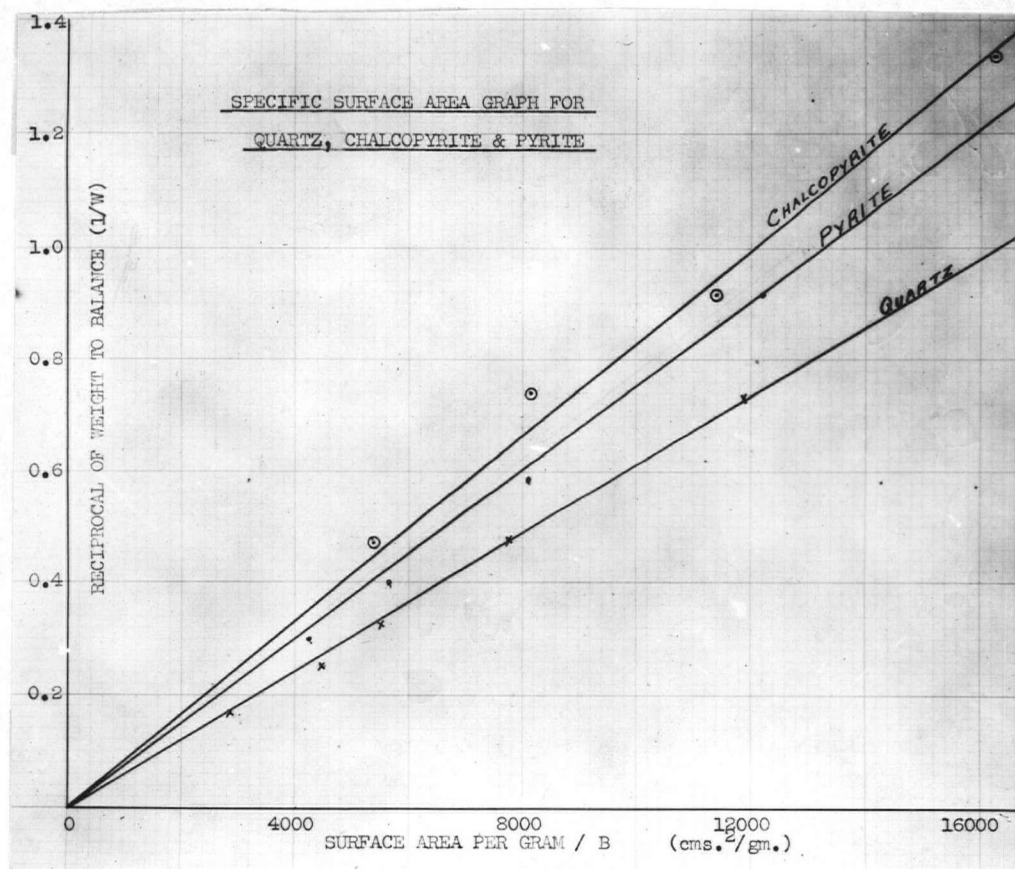


Figure 29

VI - CONCLUSIONS

The results obtained show that it is possible to determine, rapidly and accurately, the relative surface areas and average surface diameters of powders of the three minerals pyrite, quartz, and chalcopyrite, and that it is not difficult to determine the surface areas and average surface diameters of mixtures of the powders of these three minerals.

Since the physical characteristics of quartz, pyrite and chalcopyrite differ widely the results of these investigations apparently

warrant the conclusion that the photometric method of particle size measurement is applicable to mixtures of many other minerals. Certainly it seems possible that average surface diameters and relative surface areas of constant assaying materials can be directly determined from a calibration graph. Moreover, it should be possible to determine surface areas and average surface diameters of opaque materials which vary in assay. The problem becomes more difficult when two or more non-opaque materials, in varying amounts, are included in the mixture. It is suggested that a common ternary diagram might solve the problem of determining surface areas and diameters of mixtures containing two non-opaque materials, in varying amounts, besides any number of opaque materials. The apices of the triangle would represent respectively 100% opaque materials by volume, 100% non-opaque material of the first type by volume, and 100% non-opaque material of the second type by volume. Contour lines on the diagram would represent a function of the overall weight-to-balance and the average surface diameter of the mixture.

The apparatus, in its present condition, coupled with the Haultain Infrsizer will give complete size-distributions, of constant assaying materials, from approximately 75 microns down to the one micron range. With respect to surface area it seems probable that the apparatus can measure surface areas of particles down to the limits imposed by the wave length of light.

The accuracy of the apparatus and indeed the complete method of size determination appear satisfactory. Duplicate runs on the same material usually vary within less than one percent. With the finer materials one milligram is sufficient to close the electric eye from

45° to 0°.

The standard errors given in Table 1 for the calibration samples are sufficient to account for the experimental points lying slightly off the calibration lines.

Runs, such as the one given in Table IV, in which the components of a mixture are added separately to the machine to give a composite weight-to-balance reading compare within three percent or less with the calculated weight-to-balance.

In order to simplify the method of calibrating the apparatus for constant assaying mixtures the following procedure has been developed. If it is possible to assume that the calibration line for the material will be a straight line, it is necessary to determine only one point, on the particle size versus weight-to-balance graph, other than the origin, to fix the calibration line.

This point may be located by determining the weight-to-balance for a portion of the mixture which had been sized by screening. The product caught between 325 mesh and a 400 mesh screens or between 270 and 325 mesh screens would be suitable.

It would be assumed that the average particle size of the screened portion would be the mean of the openings of the screens used in the preparation. The size of the openings in the screens must be determined experimentally since prepared screens are rarely standardized.

If this method is to be used it is necessary that there be no great difference between the assay of the screened product and the assay of the initial material.

VII - ACKNOWLEDGEMENTS

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