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MINISTRY OF AVIATION

EXPLOSIVES RESEARCH & DEVELOPMENT ESTABLISHMENT

TECHNICAL MEMORANDUM No. 16/M/61

Particle Size Determination by the Sharples Micromerograph

D.J. Bullock H.C. Grant

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Particle Size Determination by the Sharples Micromerograph

by

D.J. Bullock and H.C. Grant

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17th July 1961

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Reference: WAC/117/23

1. SUMMARY

The theory and operational technique of the Sharples Micromerograph are described. An investigation has been carried out to assess its efficiency as an automated weight sedimentometer for the evaluation of weight/size distribution of particulate materials. Results show that its accuracy is reduced by failure to sediment the whole of the powder under test, this effect being accentuated in the case of most fine powders. The instrument cannot be used satisfactorily for control work unless electrostatic charging of the sedimenting material is eliminated and a modification is suggested.

2. INTRODUCTION

A survey of the methods available for the determination of size distribution of particulate substances indicates that there is a wide diversity of techniques and many different types of instrument have been developed. Certain methods, however, have been generally adopted because of modern processing requirements and/or the physico-chemical properties of the materials themselves. At present, sedimentation methods utilising either liquid or gaseous media are important, and they may be divided into three classes:

(a) turbidimetric measurement,

(b) incremental sampling,

(c) cumulative weighing.

The Sharples Micromerograph uses the third principle, the design being based upon American patents issued in 1952 and 1953 (1). Eadie and Payne (2) have developed this instrument, which is in effect an automated sedimentation balance of generous dimensions, possessing several advantages over the normal bench-type instrument. Several years ago a Micromerograph was installed at E.R.D.E., Waltham Abbey, and since then a large number of tests have been carried out to assess its efficiency as an accurate, rapid means of evaluating weight/size distributions of particulate materials.

Sedimentation can be defined as the steady fall of particles through a static medium. (The reverse process of balancing particles in an upward stream of fluid is termed elutriation.) When particles are allowed to fall under the influence of gravitational force their motion is dependent upon the difference in density between particles and fluid. They reach a constant speed, or terminal velocity, when the force exerted by the fluid drag is equal to the gravitational force. The behaviour of a sedimenting particle is also dependent upon the nature of the fluid flow produced by its own motion and varies according to particle size. Part of the resistance to motion is due to the inertia of the fluid as it is displaced by the falling particle, and part is due to the fluid viscosity. The ratio of these two forces is termed the Reynolds' Number. If this ratio is low the fluid flow tends to become virtually symmetrical around the particle. As the Reynolds' number increases, the flow becomes more turbulent and a wake develops behind the particle, involving a departure from up-and-down symmetry. If the Reynolds' number is less than 1, Stokes' law may be applied to calculate the diameter of a spherical particle settling a known distance in a given time interval:

/(Equation) .....

$$F_f = 3\pi\eta vD$$

where F_f = frictional force,

η = coefficient of viscosity of suspending medium,

v = velocity of the particle in suspension,

D = particle diameter of an equivalent Stokes' sphere

Normally, the time taken to reach a constant velocity is very small, so that the initial acceleration may be neglected and

$$v = h/t,$$

where h = fall height,

t = time,

v = velocity.

If ρ_1 and ρ_2 are the respective densities of particles and fluid, the gravitational force on each particle is

$$\frac{4\pi D^3}{3\ 8}\ (\rho_1 - \rho_2)\ g$$

When Stokes' law applies, this quantity is equal to the frictional force so that

$$3\pi\eta vD = \frac{4\pi D^3\ (\rho_1 - \rho_2)\ g}{24}$$

whence $$v = h/t = \frac{D^2\ (\rho_1 - \rho_2)\ g}{18\eta}$$

Most sedimentation methods utilise this equation since it enables the weight/size distribution of a powdered material to be calculated from a knowledge of the fractions arriving at the measurement level in known time intervals. It is advisable to ensure that the Reynolds' number is low (less than 1) by adjusting the density difference, maximum particle size and fluid viscosity. If a gas is used as the sedimentation medium, it is preferable to use modified forms of the Stokes equation. In practice most particles are irregular in shape, hence the term "particle diameter" in sedimentation work refers to the diameter of a sphere whose settling velocity and density are the same as that of the particle.

/3. .....

3. THEORY OF THE MICROMEROGRAPH

If a suspension of particles is allowed to sediment in a fluid the material collected at the base of the vessel consists of two fractions:

(a) all particles large enough to settle through the height of the suspension,

(b) smaller particles at intermediate heights.

The size/weight distribution relationship is therefore not readily deduced. However, Oden (3) and other workers have shown that graphical differentiation of the weight versus time curve will yield the size distribution by weight on a cumulative basis, and it is mainly for this reason that cumulative methods of analysis have not been readily adopted in the past. In the case of the Micromerograph the above calculation is eliminated. If we consider a suspension of particles at the top of a sedimentation column, the dimensions being such that the depth of the suspension is small compared to the length of the column, then at any given time interval all particles whose diameters are greater or equal to the Stokes diameter corresponding to that time interval will have settled out and a cumulative distribution curve on a weight basis is yielded directly. (Several workers (4, 5, 6), have utilised this principle, injecting liquid suspensions into tall liquid columns, but the formation of eddy currents makes the results unreliable.)

The sedimentation column of the Micromerograph is 85 inches long, nitrogen or air (fed from a cylinder) being used as the sedimentation medium (Fig. 1.a). The gas pressure to the head of the column is controllable up to a maximum of 400 p.s.i., the column being hermetically sealed when the instrument is in operation. The sample of powder under test (either 30 or 50 mg.) is blown by a charge of gas through a deagglomerator (Fig. 1.b) consisting of two mating cones, and thence to the top of the column where it is dispersed as a cloud of powder whose dimensions are small compared to the length of the column. For practical purposes therefore, it may be assumed that the whole of the powder under test commences sedimenting from the same horizontal plane. As previously stated, under these conditions the weight/size distribution can be calculated directly. It is important to note that, if the gas pressure in the column were less than that of the atmosphere, the powder sample would be dispersed throughout the column and the graphical differentiation already referred to would have to be used to evaluate the size distribution.

In this instrument the weight of sedimenting powder is recorded automatically on a moving chart so that the record obtained is one of cumulative weight of powder versus time. The calculation has been modified (so that the whole operation can be carried out in the minimum of time) most probably on the following basis:

Referring again to Stokes' equation

$$h/t = \frac{D^2 g (\rho_1 - \rho_2)}{18\eta}$$

/and .....

and if ρ_2 the density of the gaseous medium be neglected in comparison with that of the material under test, then

$$t = \frac{18\eta h}{D^2 g \rho_1}$$

Further, since η, h and g are constants,

$$D^2 \rho_1 = k/t$$

As a general expression, therefore $D\rho^{\frac{1}{2}} = K/t^{\frac{1}{2}}$, where D is the Stokes' diameter corresponding to the time interval t, ρ is the powder density and K is a constant. According to the manufacturers a slightly modified form of Stokes' equation is used in the case of the Micromerograph to take into account deviation from the Stokes' law drag force.

Transparent templates, graduated in values of $D\rho^{\frac{1}{2}}$ for successive elapsed time intervals are provided for superimposition on the record. In this manner the percentage accumulated weight for each successive value of $D\rho^{\frac{1}{2}}$ is determined. Dividing the latter throughout by the square root of the density of the material yields particle diameter. Since this series of quantities constitutes an oversize distribution relationship it is necessary to subtract each measured weight per cent from 100 per cent to yield the undersize distribution curve.

4. EXPERIMENTAL

4.1 Scheme of Investigation

An investigation was carried out in order to make an assessment of the Micromerograph for accurate determination of particle size distribution.

The work was mainly confined to the following:

(a) Establishment of the most representative distribution curves for a wide variety of powdered materials,

(b) The effect on measured size distribution of loss of powder to the walls of the sedimentation column,

(c) Reproducibility of results,

(d) Ability of the instrument to differentiate between samples of the same powder, each possessing slightly different specific surface areas.

A selection of some of the materials examined is given in Table 1 (p.10) together with their ranges of specific surface areas. They consisted of metallic elements, oxides, inorganic and organic compounds, in degrees of fineness varying from 780 to 1.57 x 10^6 cm^2/cm^3.

/4.2 .....

4.2 The Derivation of a Representative Distribution Curve for a Given Powder

The maximum setting of the slit width of the deagglomerator is 250 microns. This does not mean that the Micromerograph can be used to analyse powders containing particles whose size is of this order. Such particles would fall at velocities involving turbulent flow and extremely high Reynolds numbers. Furthermore, oversize particles tend to fracture or jam within the deagglomerator itself. The normal procedure is to operate the instrument with a deagglomerator setting that is twice the size of the largest particles present in the powder sample. Throughout this series of tests, each sample was carefully mixed and then examined under the microscope. Enlarged images of the prepared slides were projected on to a screen upon which had been drawn a facsimile of a ruled graticule for each magnification. All particles larger than 125 microns were removed by passing the material through a 120 B.S. mesh sieve. Ideally, the aerodynamic shear forces produced within the deagglomerator should completely disperse the sample leaving each particle intact. Preliminary runs were made using the lowest shearing force and as this force was increased to achieve complete deagglomeration the resulting analyses indicated finer distributions. These trial runs enabled the most suitable settings for each material to be established. (The manufacturers state that at 100 per cent deagglomeration, increase of the shear force does not move the distribution curve in the fine direction.)

4.3 Loss of Powder During Sedimentation

Earlier trials with the Micromerograph revealed that the magnitude of the powder losses during sedimentation was often considerable. This was mainly due to adhesion of powder particles to the wall of the sedimentation column, and it was assumed that the material had accumulated surface electric charge, the extent of this effect influencing the amount of powder collected on the pan of the servo-balance. The manufacturers of the instrument supply an antistatic agent which, when dissolved in isopropyl alcohol, can be used for coating the column wall by drawing an impregnated soft mop head through the length of the column.

In this particular investigation the materials selected were sedimented through the balance pan several times, the fraction accumulated on the balance pan being retained. When sufficient material was collected it was retested and the whole process repeated again. The size distribution curves obtained in each case were examined.

For comparative purposes size distributions were also determined by use of the photoextinction sedimentometer.

4.4 Reproducibility of Results

Particle size determination is not an exact science, but most methods are used under controlled conditions which enable the results to be fairly reproducible even if the dimensions are not absolute. In the case of the Micromerograph, which is automatically operated, the main essential is that it should yield reproducible results for a given powder using the same settings of the controls. The appropriate settings having been determined for a number of samples, the latter were then stored at ambient temperatures (between 20° and 30° C) and tested at weekly intervals.

/4.5 .....

4.5 Sensitivity of the Micromerograph in Detecting Slight Variations in Specific Surface Areas of Samples Existing in Several Grades of Fineness

Any change in the specific surface area of a particulate material must involve a change in the particle size distribution. Apparatus designed for routine size control should be able to detect small changes in specific surface area due, for example, to slight variations in processing technique or physical changes during storage. Each material examined in this particular investigation was divided into several grades of fineness, the specific surface areas differing by approximately 5 to 10 per cent when measured by an air permeability method. They were then tested on the Micromerograph.

5. RESULTS

The determination of the most representative distribution curve for a given powder was often a time-consuming operation. The manufacturers of the instrument recommend that a series of trial runs be carried out for each material, covering a wide range of deagglomerator force. It was essential to choose settings lying between those necessary to disperse the powders completely and those capable of fracturing the largest particles present. Commonly, six to eight runs were required and the choice of the correct pressure was often uncertain. The slit width of the deagglomerator was then varied in either direction and its effect on the distribution curve ascertained. The final analyses were then made using the most suitable combination of settings. In Fig. 2 are shown typical groups of curves for chromium sesquioxide and ammonium picrate, the selected curves being indicated.

Table 2 (p. 11) lists the maximum and minimum weight percentages of material sedimented on to the balance pan of the instrument for each powder. This range extends from over 90 per cent in the case of glass beads and acrylic granular resin to 6 per cent for a grade of titanium dioxide. Routine inspection of the column wall and deagglomerator housing after each run confirmed that a proportion of the powder sample had failed to sediment to the balance pan. Re-introduction of the powder fractions collected from previous runs and repeat analyses indicated a general coarsening in most cases. This effect is illustrated in Figs. 3 and 4 for aluminium powder and copper chromate. In addition, the corresponding distributions obtained by use of the photoextinction method are shown. In general, the latter indicated finer distributions.

Reproducibility tests extended over several weeks at temperatures ranging from 20° to 30°C. In the case of most coarse grade powders it was possible to obtain fairly reproducible distribution curves. Losses due to electrostatic charging, however, prevented any agreement in the results obtained for the finer grade materials. In Fig. 5 are shown results of tests carried out on ammonium perchlorate (a), and titanium dioxide (b).

The results showed that the Micromerograph could readily detect variations of 10 per cent in mean size in the case of some coarse materials possessing specific surface areas below about 6000 cm^2/cm^3. The results obtained for the finer grade powders were unreliable in most cases, and prevented any correlation between the difference in specific surface area and change in size distribution. For example, in the case of titanium dioxide, the relative positions of the distribution curves were in disagreement with the comparative magnitudes of the

/specific .....

specific surface areas. This effect is shown in Fig. 6(a). Distribution curves for coarse ground coal are also illustrated, Fig. 6(b). The values of the specific surface areas obtained by use of the air permeability method are indicated against the corresponding distribution curves.

6. DISCUSSION

The numerous tests that have been carried out with the Micromerograph on the many different types of particulate material indicate that the reliability of the instrument falls short of that required in an accurate routine sizer. In general, only a proportion of the sample under test reaches the pan of the servo-operated balance, and the manufacturers of the instrument state that this weight fraction should be "normalised" to 100 per cent for purposes of calculation. An assumption is thus made that the size distribution remains unaltered by loss of powder to the column wall during sedimentation and the collected material is taken to be identical with the original sample. Repetitive tests on pan-collected material point to a general coarsening in many cases. It is thus highly probable that the calculated size distribution is influenced by the extent of the powder loss. Reproducibility of results is often poor in the case of fine powders and numerous tests have shown that, dependent upon the fineness and physico-chemical properties of the powder being tested, losses during sedimentation can be over 90 per cent.

The elimination of the static charging of powders during testing constitutes a problem. The antistatic agent (dissolved in isopropyl alcohol) supplied by the manufacturers is only partially effective. It is probably a surface active agent whose main function is to form a continuously conducting film of moisture on the surface of the powder, enabling static accumulated charge to be dissipated more easily. Surface films of moisture form quite readily on certain materials, and decrease the surface resistivity, the magnitude of the effect being dependent upon the material. Glass exhibits this effect quite strongly and it has been found that, when the standard sample of glass beads is tested, losses during sedimentation are small.

It is sometimes possible to treat the powder under test with the antistatic agent, remove the alcohol by evaporation and leave the particles coated. Tests have been carried out on a number of substances in this way and Fig. 7 shows the distribution curves for nickel, iron and titanium dioxide powders both before and after treatment with the antistatic agent. Finer distributions were obtained in the case of the first two substances. Each series of tests described tended to lose its significance because it was found impossible to control the extent to which the materials became electrostatically charged. The factors governing the extent of this effect are complex, and in the light of present knowledge it is not clear what parameter or combination of parameters, can be utilised in order to characterise the static electrification behaviour of a given material from a quantitative standpoint, neither is it possible to bring all particulate substances to the same reference point by physical and chemical conditioning.

It has been shown by Kunkel (7) that if a powder is dispersed in air the individual particles all carry electric charges. In the case of the Micromerograph, the sample under test accumulates charge when it is blown through the deagglomerator into the top of the sedimentation column. The latter is earthed and any particle coming into contact with the aluminium surface will undergo electron transfer only at the points of contact. The contact area will be governed by the geometry of the particle and in general will be quite small.

/The .....

The time that each particle remains in contact with the column wall will therefore depend upon the electrical conductivity of the material since the particle will fall only when sufficient charge has leaked away. In the case of extremely fine particles it is possible that the van der Waals force of attraction plays a part in wall adhesion effect. The force tending to detach such particles is dependent upon their mass and is proportional to D^3 where D is the diameter of the particle. The van der Waals force, however, is proportional to D and will predominate when D is small.

Two methods of approach are available for the partial, if not complete, elimination of electrostatic charging. If a radioactive gas were a constituent of the pressurised nitrogen, the resulting ionisation would probably be effective as an aid to accelerating neutralisation. Contamination of equipment and gas leakage would, however, render this method dangerous to personnel. Beta rays are also effective and could be produced by the installation of thallium 204 foil at the top of the sedimentation column. The magnitude of the source involved would be a matter of trial and error, but it is proposed initially to install a 5-millicurie source. The use of foil is essential in order that the free fall of powder is not impeded.

Mention has already been made of the use of a modified Stoke's equation in the calculation of the values of $d\rho^{\frac{1}{2}}$. The application of Stokes' law assumes that the powder is completely dispersed and that there is no slip between the particles and the gaseous nitrogen. The latter condition is of importance only in the case of fine material, and the criterion for dispersion in the case of the Micromerograph has been discussed. However, calculations for particles larger than 50 microns in diameter settling in nitrogen indicate that the Reynolds' number is usually greater than unity. Davies (8) has developed semi-empirical equations involving the drag coefficient which yield accurate results up to a Reynolds number of 4, and it is possible that the template parameters used for data conversion are based on such relationships.

It must be emphasised that, even in its present stage of development and in spite of the criticisms already made, the Micromerograph is far superior to other automatic particle sizers (including electronic scanners with digital computation) that provide either comulative or incremental number frequencies of particles. Examination and testing of such instruments have revealed that they are still in the pre-development stage and at present unsuitable for accurate routine size analysis. The main advantages of the Micromerograph are the speed with which an analysis can be carried out, simplicity in operation, and automatic recording of the cumulative weight versus time curve.

If the proposed adaptation for reduction of electrostatic charging is successful, the Micromerograph may well represent the highest standard of achievement in the manufacture of accurate automatic particle sizing instruments. From the experience gained in using the Micromerograph, and other sizing instruments, it can be stated that every material tested must be regarded as an individual problem demanding a specific technique. No technique, or instrument, can be applied with equal success to all powdered substances.

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8. Davies, C.N., Symposium on Particle Size Analysis, Instn. Chem. Eng., 1947.

/TABLE 1 .....

TABLE 1

Material	Specific Surface Area Range, cm^2/cm^3
Acrylic granules	780 - 1060
Ground coal	1250 - 7130
Ammonium perchlorate	1770 - 9000
Standardised glass beads	2200
Aluminium powder	2900 - 137000
Micronised graphite	4000 - 41000
Ammonium dichromate	4830
Sodium carbonate	5950
Lead chloride	7390
Sand	9060
Aluminium oxide	9650
Calcium carbonate	12040
Iron powder	12340
Cobalt chromate	14600
Nickel powder	16650
Zirconium powder	20570
Red lead	29940
Titanium dioxide	61400 - 130000
Copper chromate	72560
Manganese dioxide	166600
Chromium sesquioxide	345000
Silica	1575000

TABLE 2

Material	Per Cent Sedimented Weight	
	Minimum	Maximum
Acrylic granules	80	92
Aluminium oxide	41	60
Aluminium powder	10	59
Ammonium dichromate	68	83
Ammonium perchlorate	28	53
Ammonium picrate	28	66
Calcium carbonate	24	38
Chromium sesquioxide	8	27
Cobalt chromate	57	77
Copper chormate	41	50
Ground coal	37	52
Iron powder	27	30
Lead chloride	8	31
Manganese dioxide	39	50
Micronised graphite	52	78
Nickel powder	16	36
Red lead	37	80
Sand	36	50
Silica	22	47
Sodium carbonate	38	87
Standardised glass beads	82	95
Titanium dioxide	6	41
Zirconium powder	11	51

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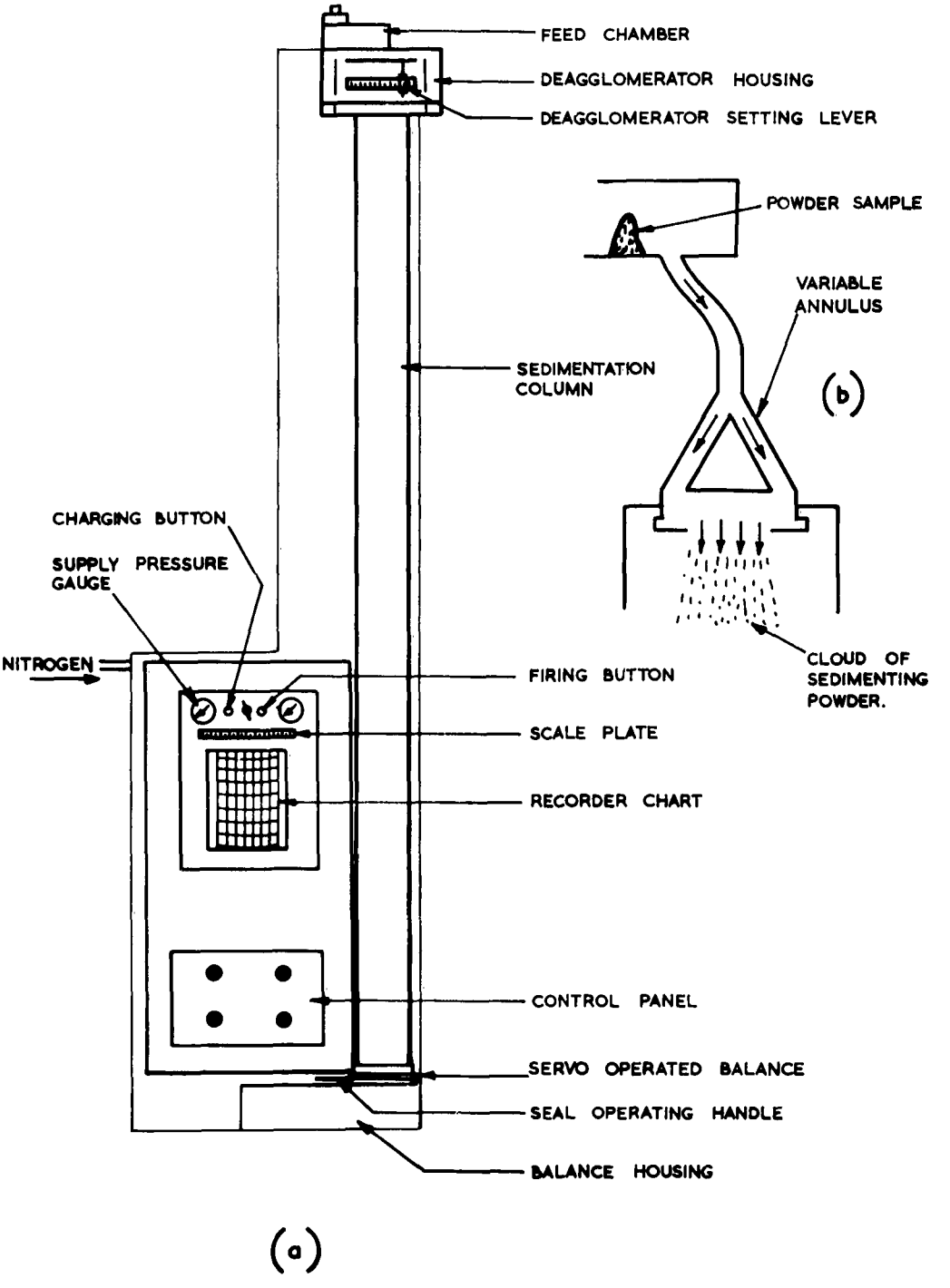
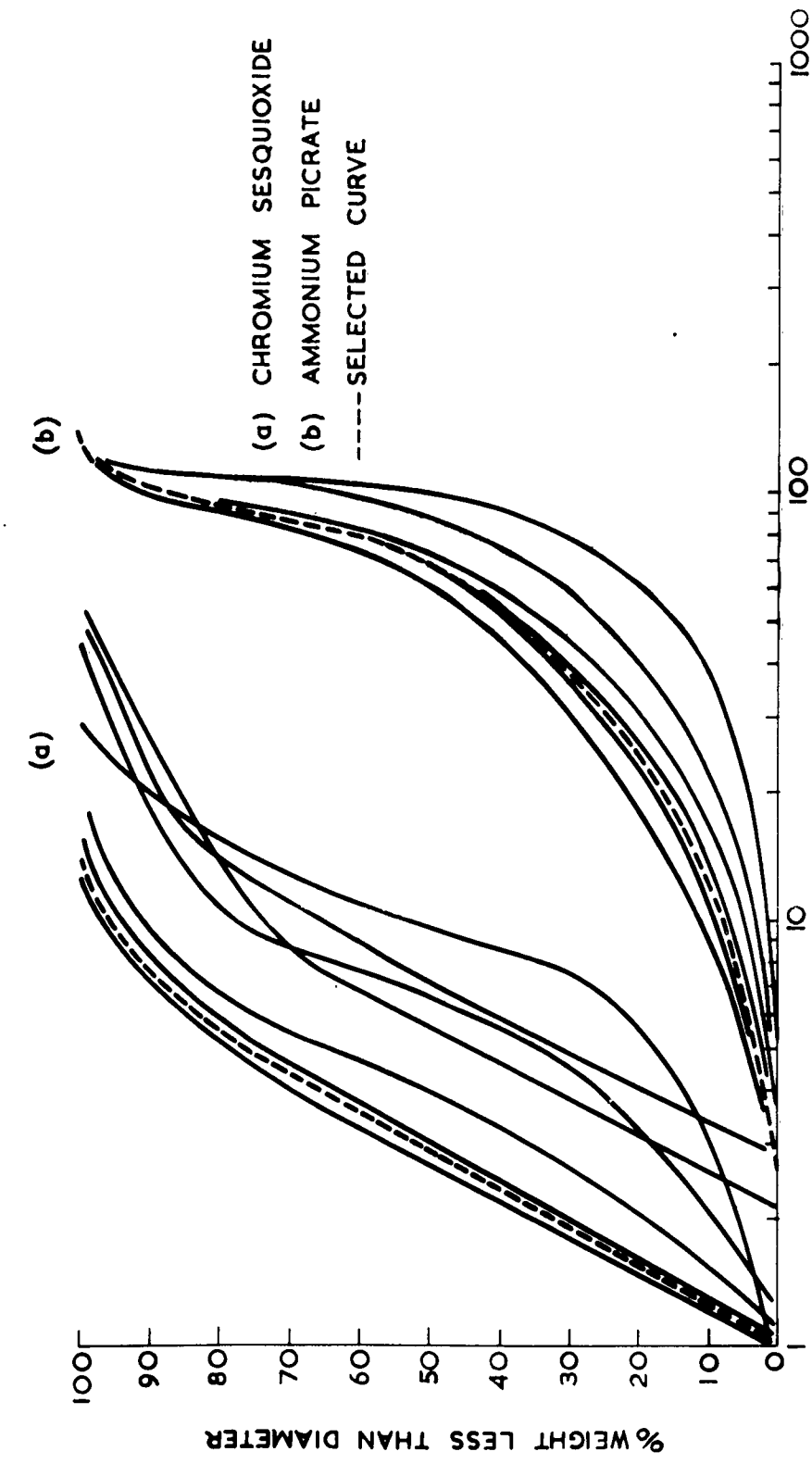
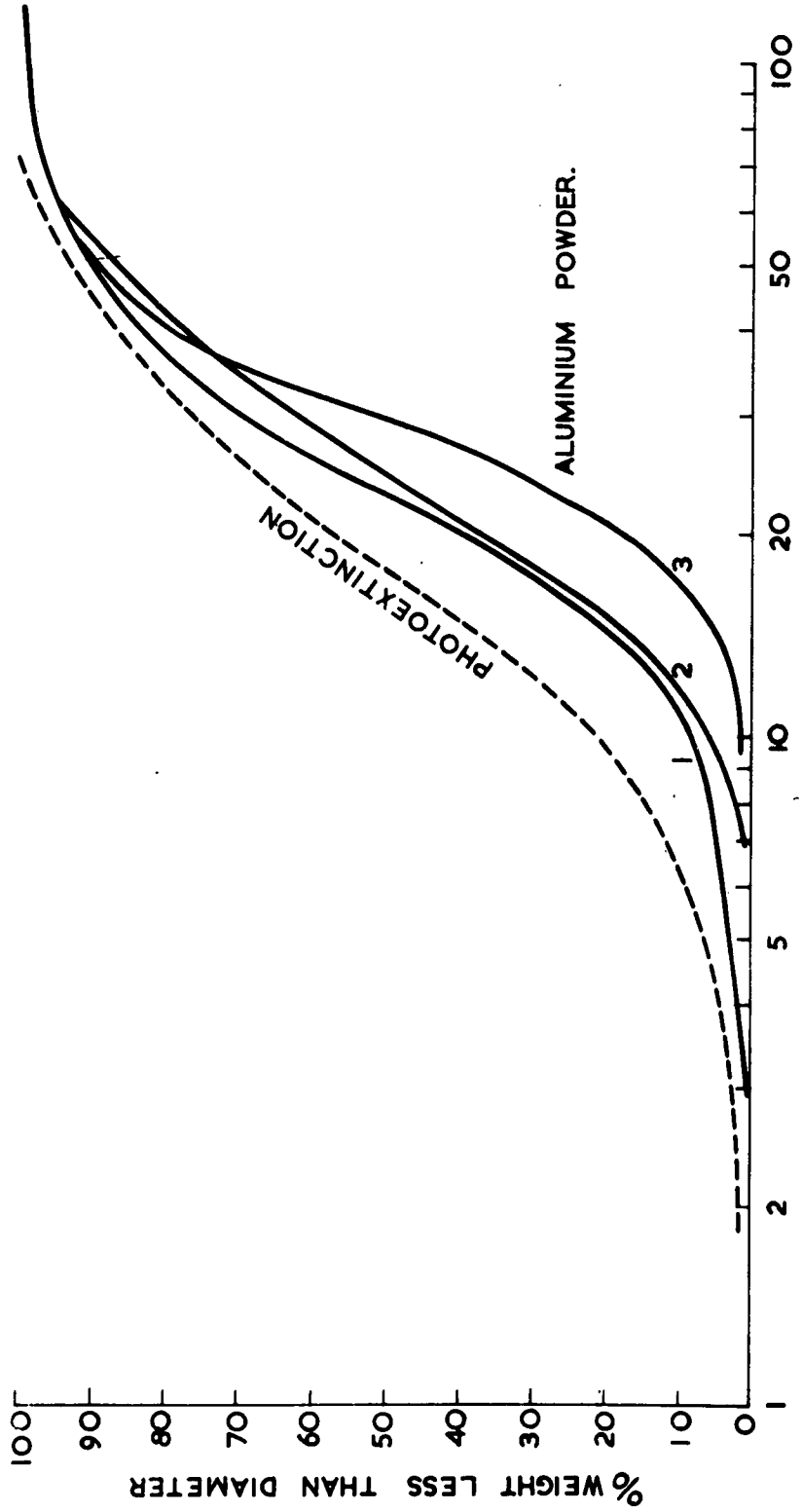

FEED CHAMBER
DEAGGLOMERATOR HOUSING
DEAGGLOMERATOR SETTING LEVER
POWDER SAMPLE
VARIABLE ANNULUS
SEDIMENTATION COLUMN
(b)
CHARGING BUTTON
SUPPLY PRESSURE GAUGE
NITROGEN
FIRING BUTTON
SCALE PLATE
RECORDER CHART
CLOUD OF SEDIMENTING POWDER.
CONTROL PANEL
SERVO OPERATED BALANCE
SEAL OPERATING HANDLE
BALANCE HOUSING
(a)

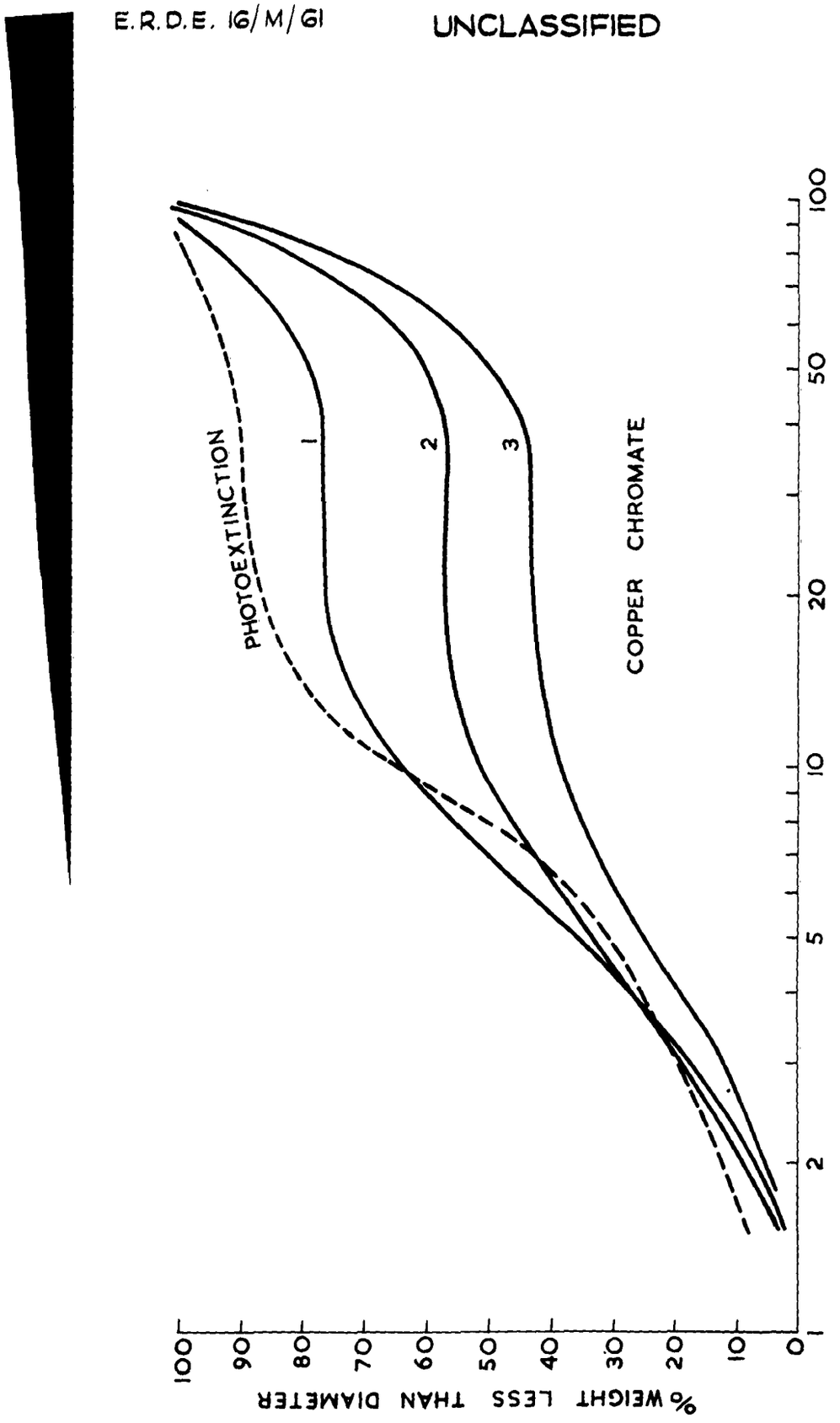
FIG. 1.



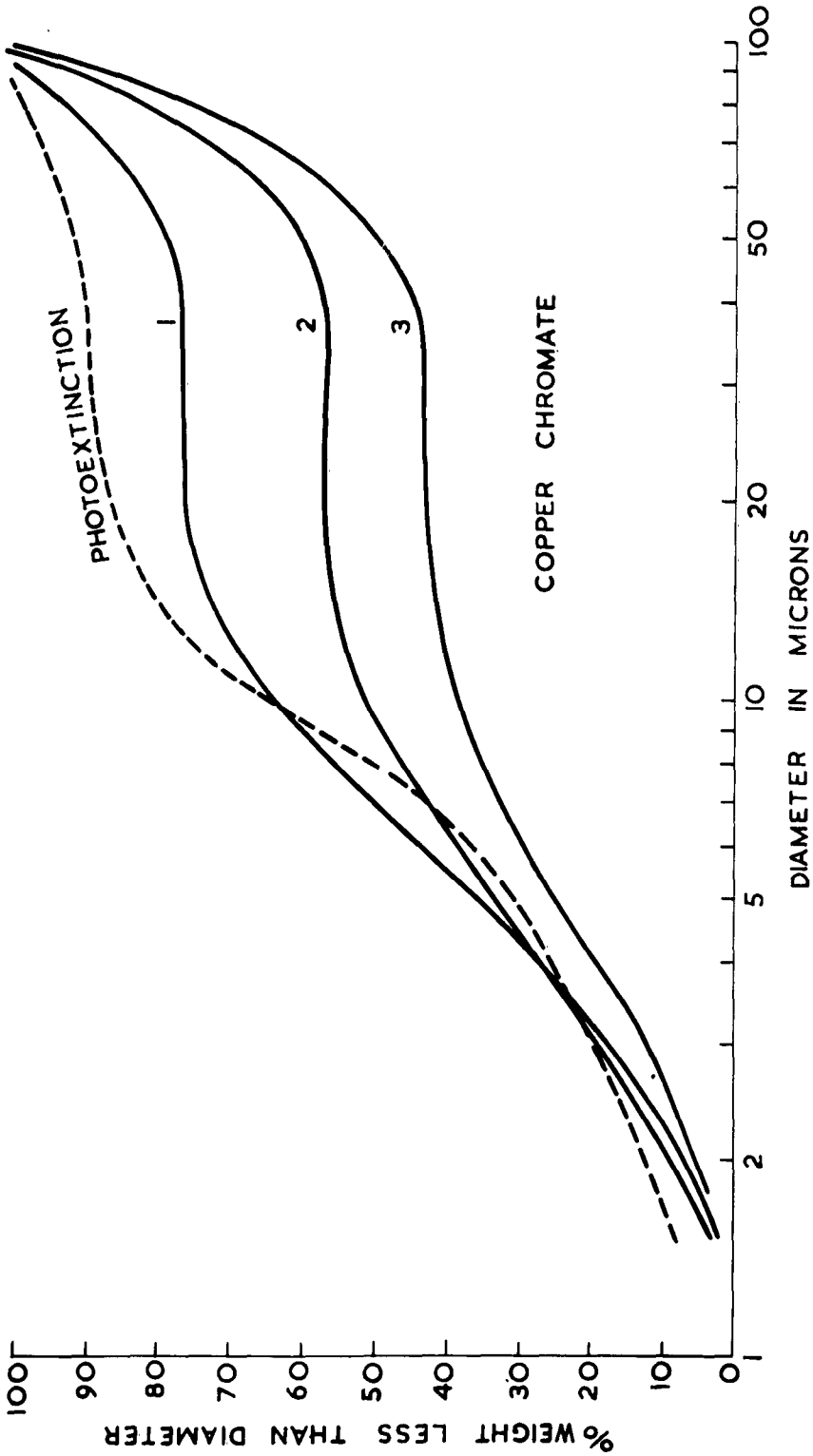
SELECTION OF DISTRIBUTION CURVE. FIG.2.



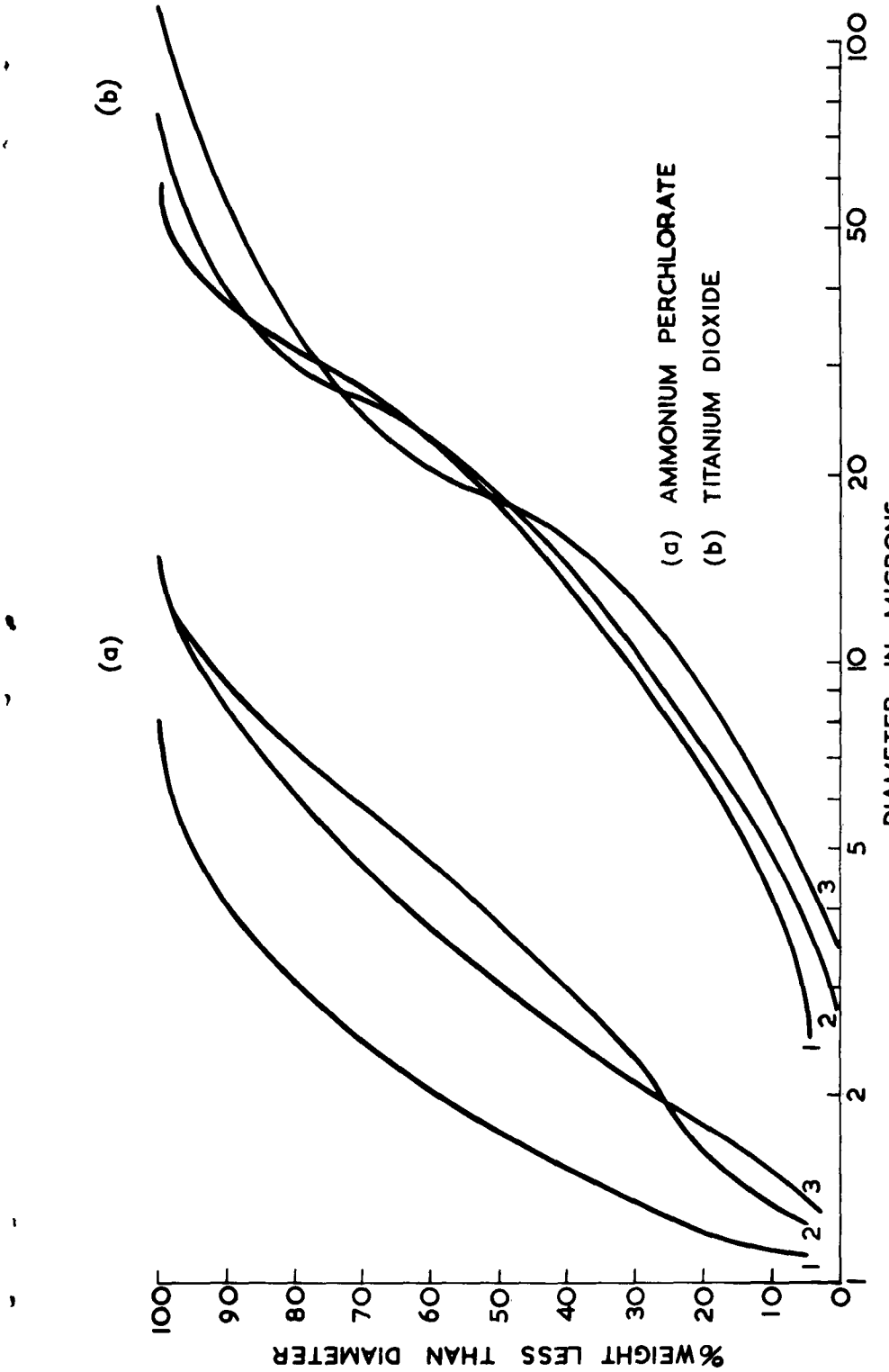
REPETITIVE TESTING ON COLLECTED MATERIAL. FIG.3.



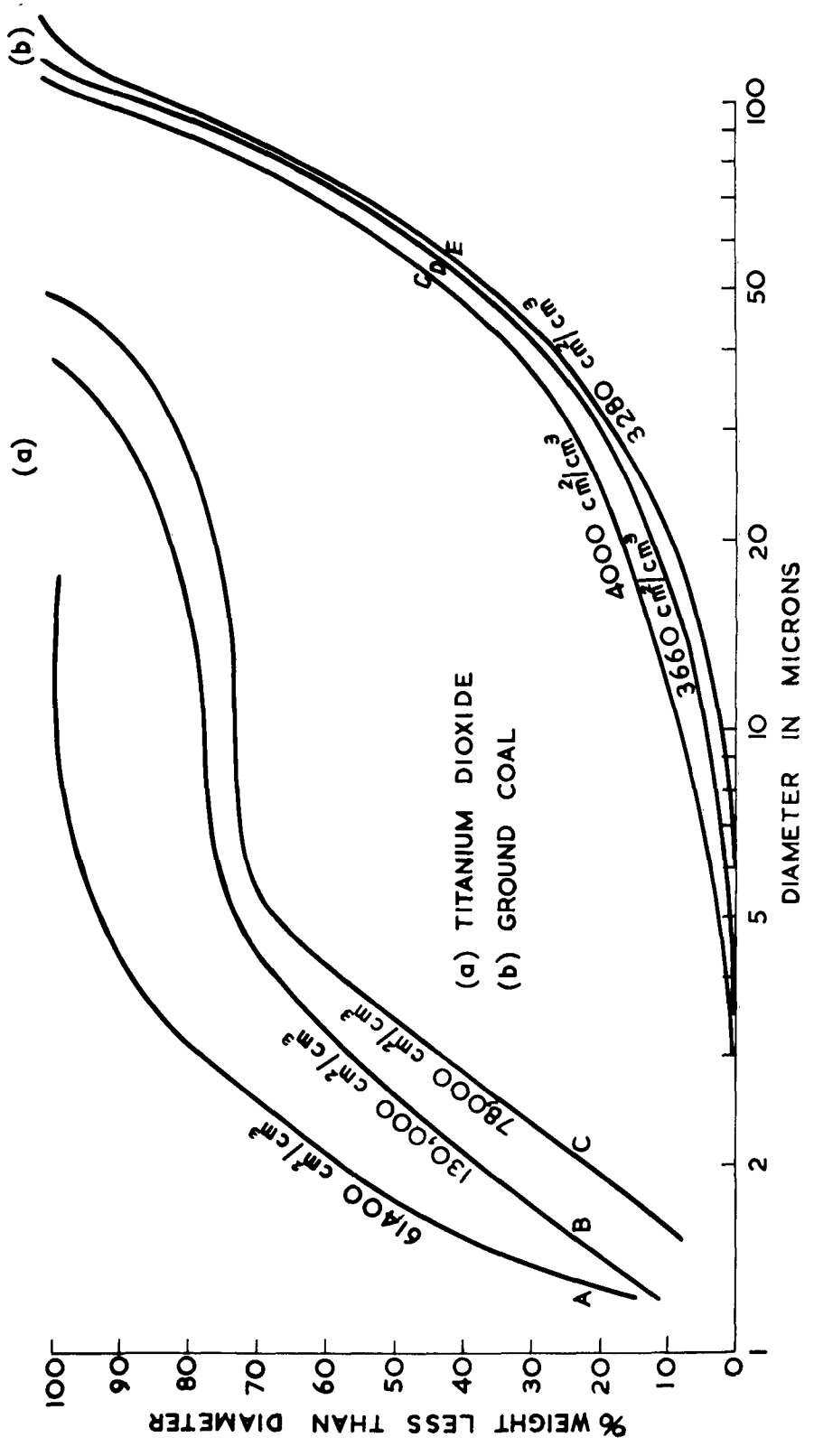
REPETITIVE TESTING ON COLLECTED MATERIAL. FIG. 4.



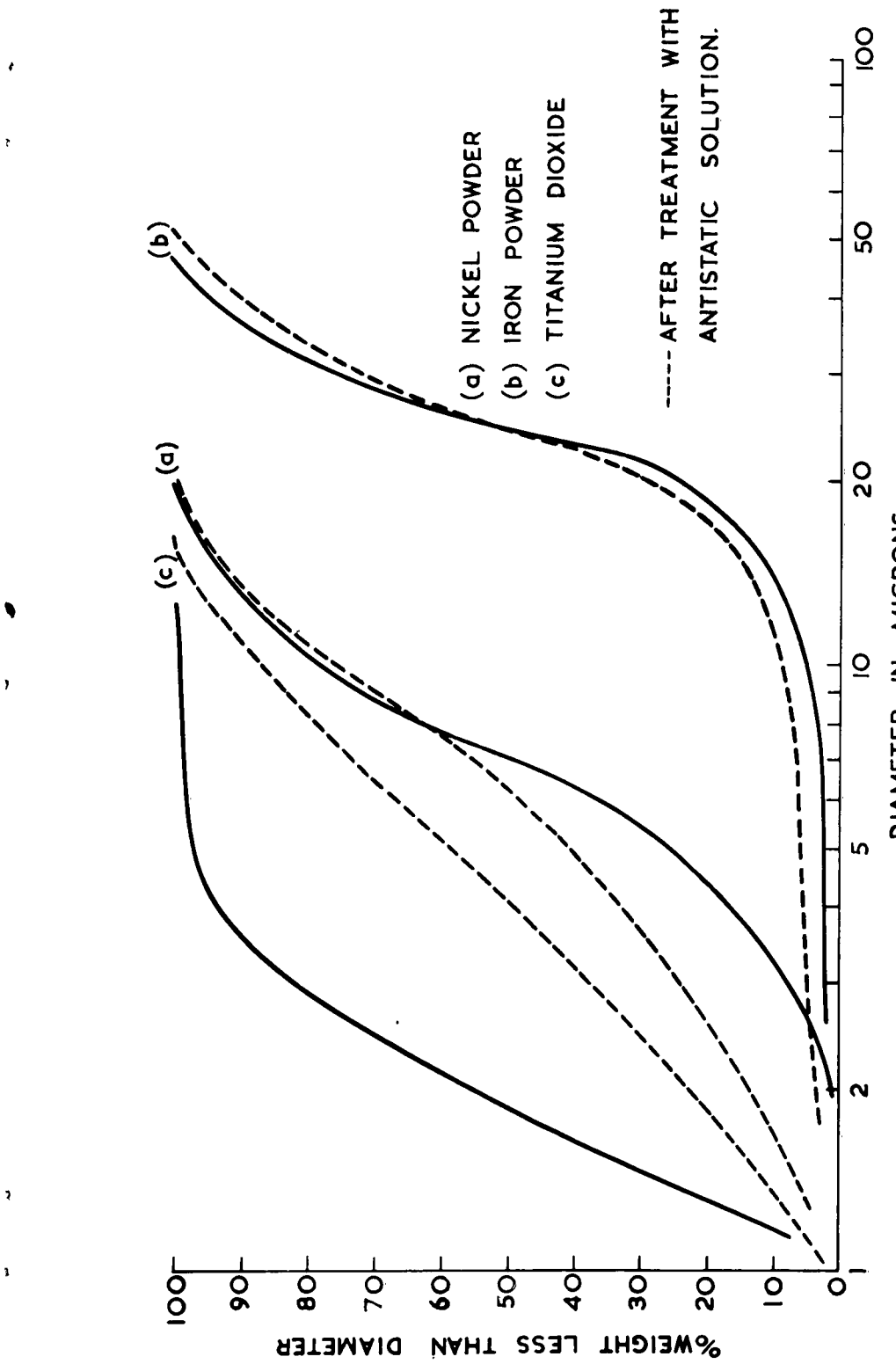
REPETITIVE TESTING ON COLLECTED MATERIAL. FIG. 4.



REPRODUCIBILITY. FIG. 5.



SENSITIVITY. FIG. 6.



TREATMENT WITH ANTISTATIC AGENT. FIG. 7.

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Particle Size Determination by the Sharples Micromerograph

D.J. Bullock and H.C. Grant Sept., 1961

The theory and operational technique of the Sharples Micromerograph are described. An investigation has been carried out to assess its efficiency as an automated weight sedimentometer for the evaluation of weight/size distribution of particulate materials. Results show that its accuracy is reduced by failure to sediment the whole of the powder under test, this effect being accentuated in the case of most fine powders. The instrument cannot be used satisfactorily for control work unless electrostatic charging of the sedimenting material is eliminated and a modification is suggested.

11 pp., 7 fig., 2 tables

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Particle Size Determination by the Sharples Micromerograph

D.J. Bullock and H.C. Grant Sept., 1961

The theory and operational technique of the Sharples Micromerograph are described. An investigation has been carried out to assess its efficiency as an automated weight sedimentometer for the evaluation of weight/size distribution of particulate materials. Results show that its accuracy is reduced by failure to sediment the whole of the powder under test, this effect being accentuated in the case of most fine powders. The instrument cannot be used satisfactorily for control work unless electrostatic charging of the sedimenting material is eliminated and a modification is suggested.

11 pp., 7 fig., 2 tables

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