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The measurement and control of
particle size in pyrotechnic ingredients

A. M. Wild

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ROYAL ARMAMENT RESEARCH AND DEVELOPMENT ESTABLISHMENT

R.A.R.D.E. MEMORANDUM (X)3/63

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The measurement and control of
particle size in pyrotechnic ingredients

A. M. Wild (X4)

Summary

The need for the control of particle size of sub-sieve powders in pyrotechnic work is stressed and methods for the measurement of specific surface and particle size distribution are discussed with special reference to the Fisher Sub-Sieve Sizer and the Sharples Micromerograph.

A brief introduction to the subject of classification of powders is followed by an account of the methods used at Langhurst to assess the efficiency of the Mikroplex Spiral Air Classifier and the Japanese Hosokawa Microseparator. Practical considerations involved in the choice of classifiers are discussed.

Preliminary investigations into methods of deagglomerating powders are briefly outlined.

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1. REFERENCES

Project Nos. 2160 and 2241A.

2. INTRODUCTION

The need for the control of particle size of sub-sieve powders used in pyrotechnic compositions has been realised for a good many years. Little, however, was done about this problem until the beginning of 1960, when the problem was examined with special reference to gasless delay compositions. This Memorandum covers the progress made during the last $2\frac{1}{2}$ years.

In the field of pyrotechnics, the use of compositions consisting of very finely divided powders, homogeneously mixed, is frequently called for. Examples are:-

- (a) For high accuracy delays, where burning rates must be uniform and strictly controlled within narrow limits,
- (b) for infra-red flares and photoflashes, where the I.R. and visible light outputs, respectively, must reach peak intensity rapidly, and
- (c) for gasless priming compositions.

In relation to such requirements, the specific surface (or mean particle size), the particle size distribution, and the shape of the particles in a composition, are all of importance.

The specific surface of the chemical components of a pyrotechnic mixture have been shown to affect the burning characteristics of such compositions. In R.A.R.D.E./X4, however, only the systems sodium nitrate - magnesium and potassium nitrate - tetranitrocarbazole have been examined and much work needs to be done on similar lines.

The effect of particle size distribution upon burning rates (for a constant specific surface) does not appear to have been investigated. It is considered that it should be. Furthermore, size distribution is an important factor to be considered when attempting to obtain a homogeneous mixture of several ingredients. To this end, there is a need to be able to produce powders with a restricted size range of, say, 5 microns.

The shape of particles in a mix affects their packing and flow properties, their ignitability and probably the burning rate - at least in the case of gasless compositions where the area of the surface in contact is particularly important.

To investigate the separate effects of specific surface, size distribution and shape, the primary requirement was undoubtedly some means for measuring the first two of these with reasonable accuracy.

3. PARTICLE SIZE MEASUREMENT

3. 1. Specific surface and surface mean diameter

The first problem to be tackled was the determination of specific surface, since the burning rates of mixtures were known to vary markedly with this factor. From a perusal of the literature and by seeking the advice of other Departments who already had considerable experience in

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the determination of specific surface, it was decided that some form of air permeability apparatus would provide fairly reproducible figures, although it was appreciated that these were unlikely to represent an absolute result especially with materials which had a high internal surface. It was considered, that provided different batches of the same material, prepared in a similar manner, had more or less the same specific surface, then they could be relied upon to behave in an identical manner in delay compositions, other things being equal.

Initially, a modified form of Rigden's apparatus, designed by D.C.I., was used for specific surface determinations and proved fairly satisfactory. Later, a Fisher Sub-Sieve Sizer, the standard instrument used in the United States, was introduced. This was found to give more consistent results, due largely to the method of packing the bed of powder and, furthermore, its use did away with the necessity for making a laborious calculation each time. Results obtained by the two methods normally agreed to within about 5 per cent. The Rigden's apparatus is still used in M_4 for coarse powders outside the range of the Fisher Sub-Sieve Sizer and in those cases where the porosity of materials is less than 0.4, when the Fisher apparatus again cannot be used. It is also used for very fine powders whose average particle size is less than 2 microns since, in such cases, it is necessary to compress the bed of powder more than is possible in the Fisher Sub-Sieve Sizer.

By the somewhat crude expedient of grinding an ingredient to a predetermined specific surface, it was possible, after the standardisation of other variables, to prepare delays, containing composition of the type potassium dichromate/boron/silicon, having a consistent burning time.

3.2. Measurement of particle size distribution

Attention was now directed towards the measurement of particle size distribution and its control in the production of powders. The primary importance of this latter factor appeared to lie in its effect upon the mixing of powders in the wet state, because segregation would occur very readily in powders having a wide range of particle sizes. Although, in this country, mixing is mostly carried out in the dry state or in the form of stiff pastes, segregation in dry mixes still occurs to an appreciable extent. Quite apart from this, however, it was considered (as indicated above) that variation in particle size distribution for a fixed average particle size might itself have an independent effect upon burning rates, and it was desired to investigate this point. Furthermore, a powder may be completely specified by means of its particle size distribution curve whereas in the case of a specific surface this is not so.

A Roller Analyser was purchased from the United States both for particle size distribution studies and with the idea of preparing samples of powder with very limited size ranges such as 5-10 microns, 10-15 microns and 15-20 microns. It soon became evident that while this instrument was effective for metal powders like aluminium, it was useless for dealing with fine particles of materials which agglomerate readily. Far too much time is necessary for a single analysis, a separate determination being necessary for each point on the curve.

Based on the experience gained by Mr. Grant of E.R.D.E. and from other sources, it was concluded that for the measurement of particle size distribution, there is probably no better equipment at present available than the Sharples Micromerograph.

The Micromerograph determines the particle size distribution of fine powders down to about 1 micron. It works on the principle of sedimentation in compressed nitrogen. The sample for analysis is forced, by means of a blast of dry nitrogen, through two adjustable mating cones, thereby becoming deagglomerated. It then falls through a column of dry compressed nitrogen on to the pan of a continuously recording balance. From a knowledge of the density of the sample, the cumulative weight versus time curve traced out by the instrument is converted, by Stokes' Law, (using a template) to a cumulative weight versus particle diameter curve. The Micromerograph has the marked advantage over other sedimentation methods of operating in a dry gas, so that water soluble materials present no

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no difficulty. Also, even the finest particles will settle in a few hours whereas, with sedimentation in liquids this would take several days.

E.R.D.E.'s opinion of the instrument is summarised in the concluding two paragraphs of a recent Technical Memorandum: (1).

"It must be emphasised that even in its present stage of development and in spite of the criticism already made, the Micromerograph is far superior to other automatic particle sizers (including electronic scanners with digital computation) that provide either cumulative or incremental number frequencies of particles. Examination and testing of such instruments has revealed that they are still in the pre-developmental 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. On the experience gained 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".

In a recent report (2) Picatinny Arsenal, while drawing attention to the limitations of the Micromerograph, reinforced the opinion of E.R.D.E. by stating that:-

"the Sharples Micromerograph is suitable for control purposes where reproducibility of results is of prime importance",

E.R.D.E. have recently reported a substantial improvement in the performance of the Micromerograph, particularly for very fine powders, an area in which X4 have a particular interest. This has been achieved by the combined use of:-

- (a) a radioactive thallium lining to the settling chamber*, and
- (b) the addition of 1 to 2 per cent of finely powdered graphite to the sample.

Both these devices are designed to reduce static charge, the existence of which tends to cause agglomeration and also to prevent some of the material from settling.

4. PARTICLE SIZE CLASSIFICATION

4.1 General survey

Pending the availability of a micromerograph at X4, it appeared desirable to look into the question of particle size classifiers for the production of powders within prescribed particle size ranges.

A thorough search was made of the various classifiers available on the market. Any type of classifier utilising a liquid for separation of particles was ruled out since some of the materials for gasless delay compositions are water soluble or affected by water and, furthermore, such separations may be very lengthy when the particles are extremely fine. There remained centrifuges operating in gases or gas elutriators, neither of which can produce clear cut fractions. However, two machines were found which worked on the principle of opposing centrifugal force by air flow. These are the Japanese Hosokawa Microseparator and the German Mikroplex Spiral Air Classifier. By way of explanation, in very broad terms, it may be said that if particles of various sizes are flung outwards from a source

*Now discontinued

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by centrifugal force, the larger particles will travel furthest whilst in an air stream, the reverse is true. If then an air stream is made to oppose the motion of particles imparted by centrifugal action, particles of one particular diameter will be equally affected by the two forces (depending upon their relative magnitude). Such particles will remain stationary whilst smaller ones go in one direction and larger ones in the other. By changing the relative strength of the two forces, a powder may be "cut", sharply, at any required particle diameter - at least, theoretically.

4.2 Assessment of Classifiers

With a view to comparing the relative efficiency of the Japanese and the German classifiers, both Alpine (the manufacturer of the Mikroplex classifier) and the Japanese firm were asked to classify a sample of potassium dichromate provided, into the fractions 0 to 5 microns, 5 to 10 microns, 10 to 15 microns, 15 to 20 microns. This material was selected because it promised to be a difficult one to classify. Alpine delivered a 0 to 6 micron sample stating that separations of the type 5 to 10, 10 to 15, 15 to 20 μ etc. were not possible because of agglomeration of particles. The Japanese firm (The Hosokawa Ironworks Ltd.) did not attempt to supply what was asked for but merely "out" the sample at 20, 15, 10 and 5 microns, thus producing fractions of the type 0 to 5, 0 to 10, 0 to 15, and 0 to 20 microns. Later on, however, when they had more time, they cut a 0 to 20 micron sample at the 10 micron level. The fine fraction was again cut, this time at the 5 micron level and the following four samples, accompanied by photomicrographs, were sent to us:-

- (i) Raw material, 0 to 20 microns
- (ii) A coarse fraction approximately 10 to 20 microns
- (iii) A fine fraction, 0 to 10 microns
- (iv) A still finer fraction, 0 to 5 microns

The efficiency of the Japanese classifier was assessed in the following way:-

- (i) By means of the photomicrographs supplied
- (ii) By carrying out a particle size distribution analysis on all four samples on E.R.D.E.'s Micronerograph
- (iii) By determining the average particle size (surface mean diameter) of the samples on the Fisher Sub-Sieve Sizer. Finally, in the case of the 0 to 5 micron sample, the average particle size was calculated from the size distribution curve. By this means relationships between the microscopic data, the Micronerograph, the Fisher Sub-Sieve Sizer and the classifier could be established.

4.3 Results and discussion

Photomicrographs of the four Japanese samples are shown in Fig.1 of the Appendix. From the accompanying micron scale, the separations appear to have been effective, and this is borne out by the four corresponding size distribution curves obtained on the Micronerograph, shown in Fig.2.

Fig. 3 is a histogram showing the particle size distribution by number of the 0 to 5 micron sample obtained from the curve (a) in Fig.2. In Fig.4 this histogram has been converted to a cumulative particle size distribution by number curve. From this cumulative curve, it can be seen that approximately 90% of the particles have a diameter of less than 5 microns, in excellent agreement with the photomicrographs.

Lastly, when the average particle size was calculated from the histogram, a figure of 3.0 microns was obtained, in very fair agreement

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with the figure of 2.7 obtained on the Fisher Sub-Sieve Sizer. An attempt to get agreement between the average particle size of the 10 to 20 micron fraction as determined on the Fisher, and as derived from the micromerograph, was disappointing, these values being 12.4 microns and 5.4 microns respectively. The reason for this is not clear.

The German Mikroplex Spiral Air Classifier could not be assessed in the same way as the Japanese instrument since only a single fraction was available for analysis. However, the size distribution curve obtained from a nominal 0 to 6 micron fraction was almost identical with that obtained for the 0 to 5 micron fraction prepared by the Hosokawa Microseparator. This seems to indicate that the efficiency of the two classifiers is approximately the same and, since both operate on the same principle, it is not unreasonable to assume that their performances would be similar for the coarser cuts.

Although, in order to assess the classifiers properly, much more work along the lines indicated above was desirable, this was not possible because the manufacturers could not be expected to devote any more time to classifying further samples for experimental purposes.

5. PRACTICAL CONSIDERATIONS IN THE CHOICE OF CLASSIFIERS

Alpine stated that great difficulty was experienced when dealing with potassium dichromate because, due to agglomeration, the narrow feed pipe of the Mikroplex rapidly became blocked. They suggested however, that a wire passing through the pipe and attached to a vibrating plate within should solve this problem. This classifier occupies little space and is simple to clean.

The Hosokawa Iron Works did not make any mention of blockage of feed pipes (although it may well have occurred) but they did complain strongly about the difficulty of "ridding the machine of this truly contaminative material". There is no doubt that the cleaning out of the Hosokawa Separator would be time consuming.

Another point to be borne in mind is that of servicing. Alpine are well represented in London by their agents Lavino and, if need be, problems could be referred readily to the manufacturer in Augsburg. On the other hand, although the Japanese firm have an agent operating close to X4 the impression gained is that he would have little time to devote to after sales service and if any serious problems arose they would have to be referred to the manufacturer in Japan.

It is considered that if the feed pipe blockage problem in the Mikroplex Classifier admits of the simple solution suggested, then it would be preferable to purchase the German classifier.

6. DEAGGLOMERATION OF POWDERS

This problem has received limited attention. The use of a low viscosity silicone for surface coating certain fine powders has been investigated by C.D.E.E., Porton, who found that, in some cases, powders could be effectively dispersed. Attempts in X4 to apply this method to potassium dichromate were not successful.

A second approach was the addition of half to one per cent of finely divided silicates or silica to the agglomerated powders. Such anti-caking agents marketed by Joseph Crosfield & Sons Ltd., Warrington, under the names Microcal, Gasil 200, Neosyl, Alusil, etc. are somewhat selective in action and are used commercially for de-caking materials such as common salt. None of these anti-caking agents appeared to reduce the tendency of potassium dichromate to agglomerate, but, in a series of tests carried out

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for X4 by the supplier, it was concluded that caking of dichromate under pressure could be reduced from 74% to 32% by the admixture of 1% of either Gasil 200 or Gasil 23. In X4 it was found that 0.5% of Neosyl would render barium nitrate free flowing. These approaches, therefore, are worth considering further.

7. CONCLUSIONS

7.1 The control of particle size of sub-sieve powders in pyrotechnic work is of considerable importance.

7.2 For the routine determination of average particle size down to 2 microns the Fisher Sub-Sieve Sizer is the most suitable equipment. Powders finer than this must be compressed more strongly than is possible in the Fisher apparatus, and their average particle size may be determined in a Rigden's apparatus.

7.3 The Sharples Micromerograph is the best equipment available for routine size distribution analysis where reproducibility is the main consideration. The Roller Analyzer has been found to be of limited application and in use is very time consuming.

7.4 For the classification of powders, the German Mikroplex Spiral Air Classifier and the Japanese Hosokawa Microseparator are probably the best instruments available. The German machine is more compact and much easier to clean between runs, but the feed pipe may rapidly become blocked with certain types of very fine powders. This problem should not be difficult to solve.

7.5 The problem of deagglomeration of particles is an important one. Mechanical methods may be useful immediately before feeding a powder to a classifier, but methods involving the use of silicones and silicates, and possibly other agents, must be studied further as a means of preventing reagglomeration after classification.

Acknowledgments

Thanks are due to Mr. H. C. Grant of E.R.D.E. for carrying out Particle Size distribution analyses for us on the Micromerograph at Waltham Abbey.

Bibliography

1. Bullock, D. J. and Grant, H. C., E.R.D.E. Technical Memorandum No.16/M/61 (1961) Particle Size Determination by the Sharples's Micromerograph
2. Kaye, S. M., Middlebrooks D.E. and Weingarten, G., Feltman Research Laboratories (Picatinny Arsenal), Technical Report FRL-T.R.-54 (1962)

Description of the Figures

Fig. 1.

Photomicrographs of potassium dichromate classified in the Japanese Hosokawa Microseparator, M.S.O.:

- (a) Material, nominally 0-5 μ diameter, obtained by cutting a 0-20 μ fraction first at 10 μ and then cutting the 0-10 μ fraction so obtained at 5 μ .
- (b) Material, nominally 0-10 μ , obtained as the finer fraction by cutting the 0-20 μ starting material at 10 μ .
- (c) Starting material, nominally 0-20 μ , obtained by cutting a ground sample of potassium dichromate at 20 μ and taking the finer fraction.
- (d) Material obtained from (c) by cutting at 10 μ and taking the coarser fraction.

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Fig. 2

Particle Size Distribution Curves by Weight, of the samples in Fig. 1, obtained with the Sharples Micromerograph.

N.B. When relating Figs. 1 and 2, since the photographs are judged by eye on a number basis, allowance must be made for the fact that the weight of a particle is proportional to the cube of its diameter.

Fig. 3.

Histogram showing Particle Size Distribution by Number of a nominally 0 - 5 μ sample of potassium dichromate. This was calculated from the curve (a) in Fig. 2.

Fig. 4

Cumulative curve for Particle Size Distribution by Number of a nominally 0 - 5 μ sample of potassium dichromate. This was obtained from Fig. 3.

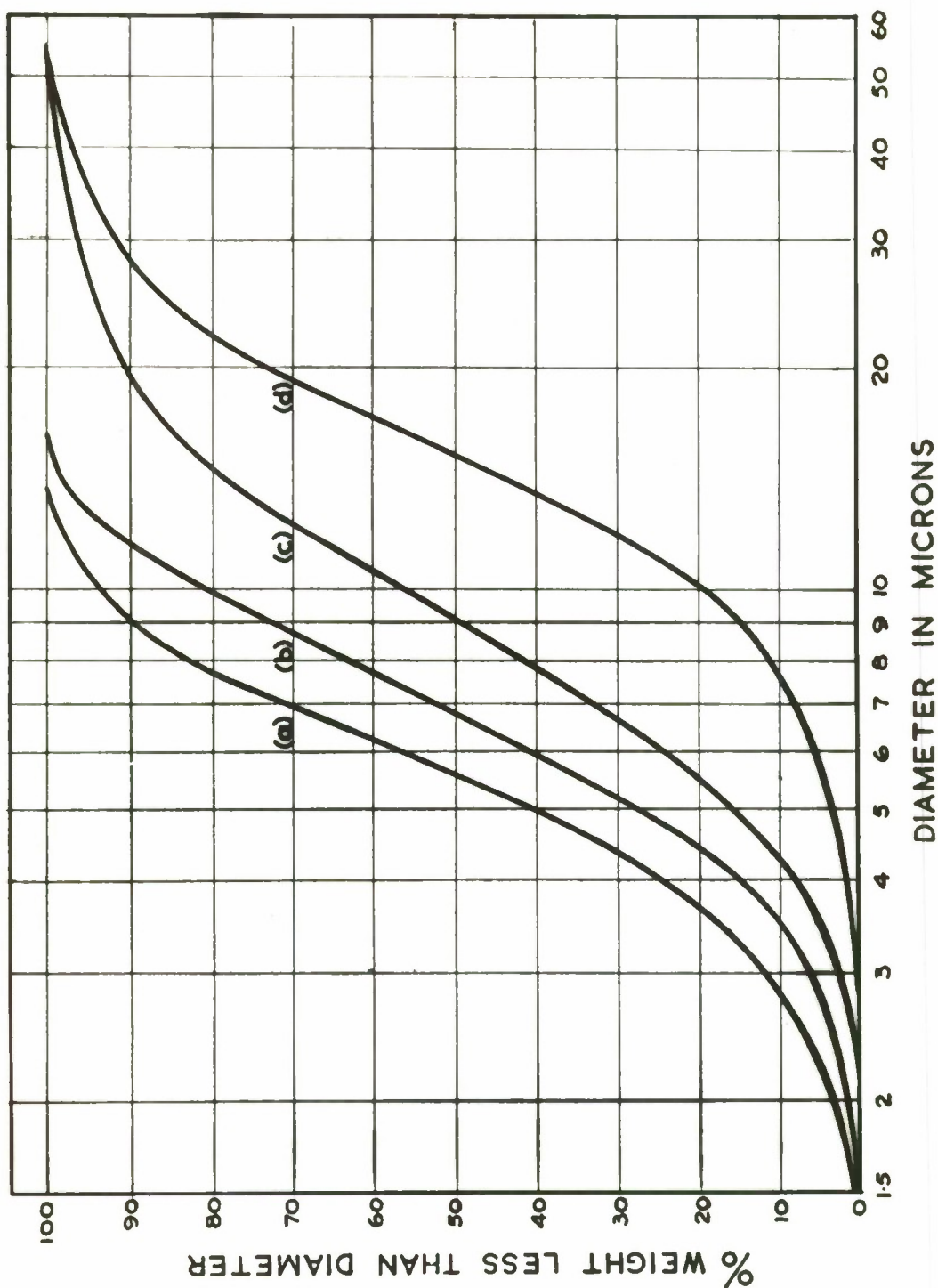
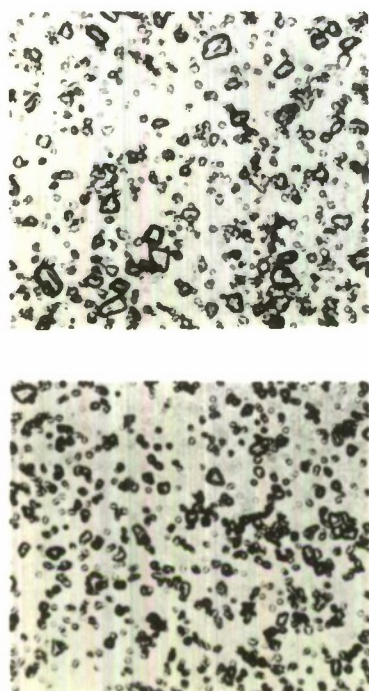
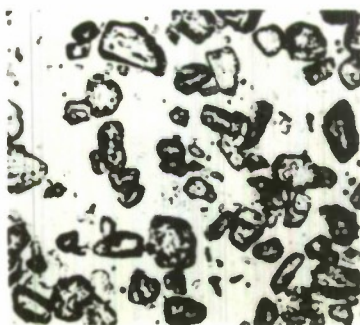


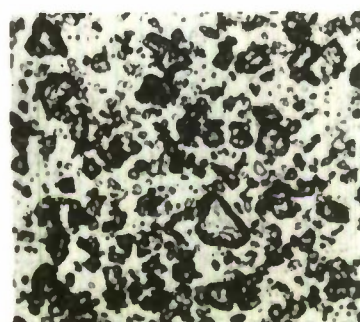
FIG.2. SIZE DISTRIBUTION CURVES FOR
SAMPLES IN FIG.1.



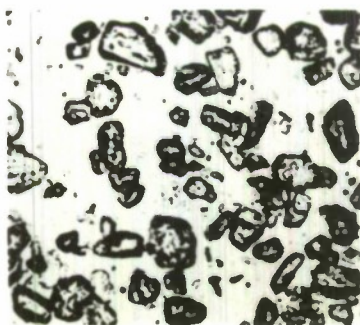
(a)



(b)



(c)



(d)

SCALE - MICRONS
0 25 50 75 100 μ

FIG.1. PHOTOMICROGRAPH (X 520)
OF CLASSIFIED POTASSIUM
DICHROMATE

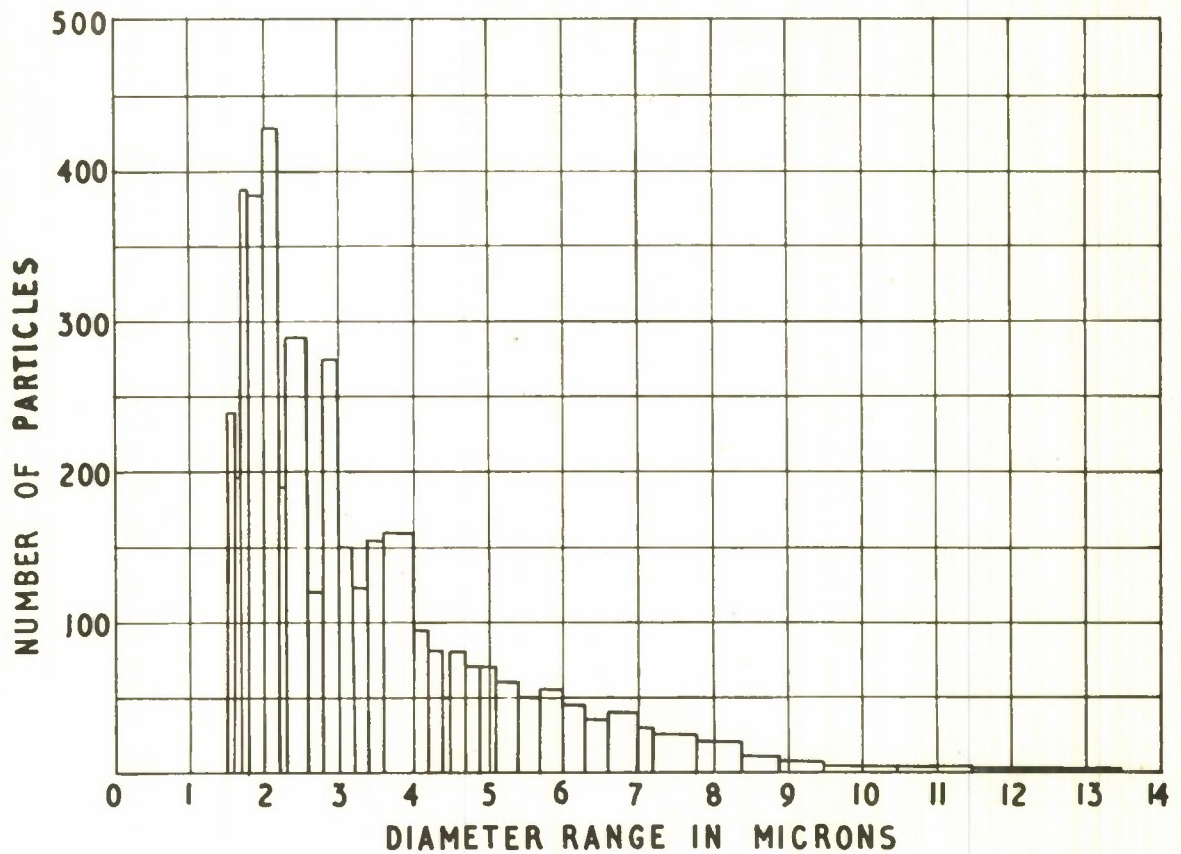


FIG.3. HISTOGRAM OF PARTICLE SIZE DISTRIBUTION
BY NUMBER DERIVED FROM CURVE (a)
IN FIG.2.

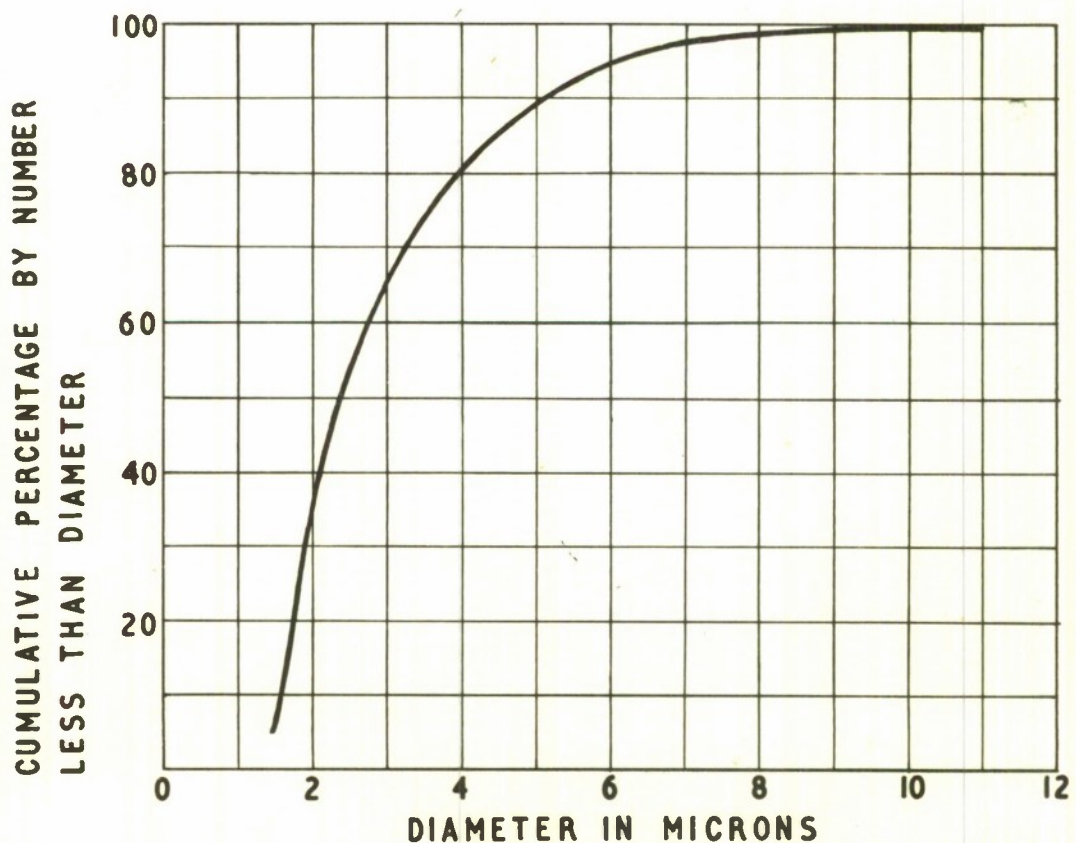


FIG 4. CUMULATIVE PARTICLE SIZE DISTRIBUTION
BY NUMBER DERIVED FROM HISTOGRAM IN FIG.3.

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A.M.Wild.

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7pp, 4 figs. 2 refs.

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