

NRL Report 6274

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The Measurement of Particle Size of Fire Fighting Dry Chemicals

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CONTENTS

Abstract	ii
Problem Status	ii
Authorization	ii
SPECIFIC SURFACE AND PARTICLE SIZE MEASUREMENTS	1
SPECIFIC SURFACE AND SIZE MEASUREMENTS	2
DISCUSSION OF MEASUREMENTS	7
CONCLUSIONS	11
RECOMMENDATIONS	12
ACKNOWLEDGMENTS	13
REFERENCES	13
APPENDIX – Methods for Particle Size and Pore Size Determination	14

ABSTRACT

Four samples of potassium bicarbonate base fire extinguishing powders, "Purple-K-Powder," which had been ground to different degrees of fineness were analyzed for particle size characteristics by several different methods. These were the Blaine Fineness test, the Roller Particle Analyzer, the Coulter Counter, surface area by nitrogen adsorption, and mercury porosity. Comparisons were made of the results of the procedures. Highest values for specific surface were obtained by the nitrogen adsorption method followed by the Blaine, the Coulter, and then the Roller Methods, in that order. No conclusions were made as to which method gave the most accurate result for particle size, but it was found that the mean diameter was a better basis for comparing results than was the median diameter. Although the relationships of specific surface values among methods were non-linear over wide ranges, calibration curves were made to facilitate conversion. It was also concluded that specific surface values, as obtained by permeability with the Blaine method, were adequate for describing the desired size of particles for fire extinguishing purposes.

PROBLEM STATUS

This is an interim report; work on the problem is continuing.

AUTHORIZATION

NRL Problem C08-15
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UNCLASSIFIED

THE MEASUREMENT OF PARTICLE SIZE OF FIRE FIGHTING DRY CHEMICALS

SPECIFIC SURFACE AND PARTICLE SIZE MEASUREMENTS

As a part of the continuing study on particle size requirements and methods of size analysis of Purple-K-Powder, KHCO_3 based, dry-chemical extinguishing agent, one of the manufacturers of this powder provided NRL with four samples in October 1964. These especially prepared samples represented different grinding batches of widely varied fineness and had been carefully analyzed in the manufacturer's laboratory by the Blaine Fineness Tester (ASTM C-204) and Roller Particle-Size Analyzer (ASTM B-293) techniques. It was suggested that NRL also analyze these samples independently as a check on procedures. The initial results were withheld from NRL to ensure independent findings.

In addition to expressing a desire for cross checking the particle size results of the four samples, the manufacturer questioned the validity of the Blaine technique as presently set forth in MIL-F-22287. The basis for raising this question was the poor relationship between specific surface (S_w) as determined by his Blaine procedure and the average particle diameter as determined by his Roller procedure. (His "average" diameter was the median size diameter.) The results are summarized as follows:

Sample	S_w (cm^2/g)	Median Diam. (microns)
a	2770	24
b	2775	39
c	3280	18
d	5440	22

It can be seen that these do not follow the relationship of the smallest particle size having the greatest specific surface.

Because of past experiences in conducting particle size determinations with the Roller device showed the results to be unreliable, NRL was not prepared to accept fully these findings without some further investigation and study. A Roller instrument was not available for making check runs at NRL, although one has since been obtained but is not yet in operation. In addition to conducting Blaine tests, steps were taken to send out portions of the four original samples for still other independent analyses using additional techniques. One method was the Coulter, a technique based on sensing electrolyte conductivity changes. This method gives a complete size distribution. The second method was nitrogen adsorption, which gives pore size area, pore size distribution, and B-E-T area.

At the present time it is difficult to state exactly what parameter regarding particle size is the most significant in the preparation of an efficient firefighting powder. On the basis of results obtained with laboratory experimental flame suppression apparatus, specific surface appears to be very important. This would seem to be valid from the chemical reaction kinetics believed to be involved during the extinguishing process (1,2). In practical extinguishment considerations, however, large particles are needed to achieve powder stream reach and this works against having a high specific surface. Presumably, a wide range of particle sizes is beneficial in order to promote non-caking properties and free flowability, and efficient particle arrangement is needed to achieve a high bulk density.

Sufficient data are not available to ascertain exactly what is optimum. Meanwhile the determinations of particle size continues to be a center of scientific endeavor and controversy.

SPECIFIC SURFACE AND SIZE MEASUREMENTS

The first step by NRL was to run Blaine analyses on the four samples. Figure 1 presents a comparison of the NRL and manufacturer's data. The ideal comparative line is indicated and reveals that the analyses, as reported by both parties, were in very good agreement. Figure 1 does not seem to indicate any problem in technique or in obtaining good comparable results between laboratories. The greatest difference between NRL results and the manufacturer's results on any of the four samples was 4%.

The Blaine method yields only a single value for specific surface and can give no information as to the range and distribution of particle sizes. From this value for specific surface, however, it is possible to calculate what the particle diameters would be if they were all the same size. This diameter is known as the mean diameter d_{vs} and is determined by the following relationship:

$$d_{vs} = \frac{6 \times 10^4}{S_w \rho} \text{ (microns)}$$

where: d_{vs} = mean dia (microns)

S_w = specific surface (cm^2/g)

ρ = density (g/cc)

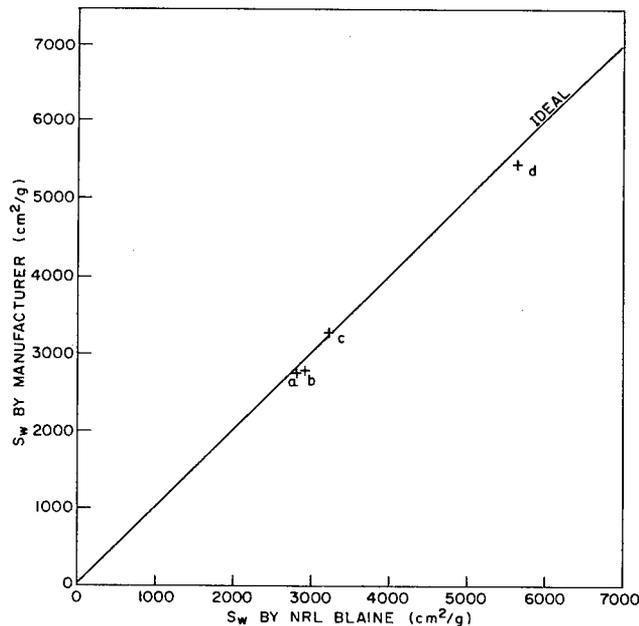


Fig. 1 - Specific surface comparison by Blaine Analysis run by NRL and manufacturer

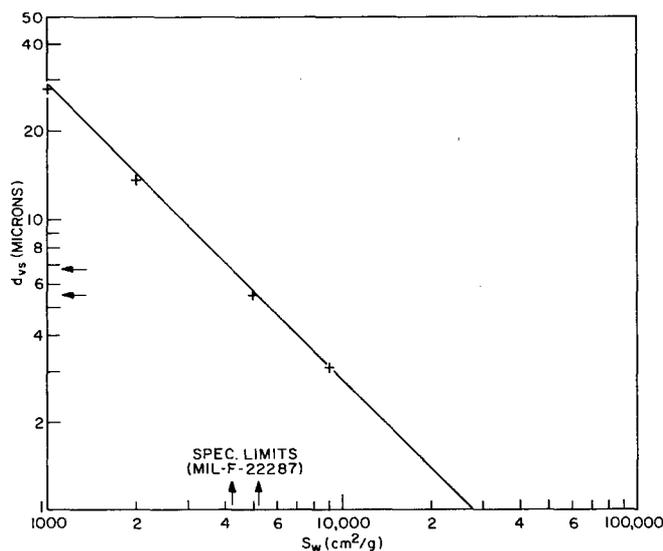


Fig. 2 - Conversion of specific surface to mean diameter

Using 2.17 as the density of the powder, this equation becomes

$$d_{vs} = \frac{2.77 \times 10^4}{S_w} \text{ (microns) .}$$

A plot of this equation is given in Fig. 2 with the specific surface specification limits of 4200 to 5200 cm²/g indicated. These limits correspond to mean particle diameters of 5.5 and 6.9 microns.

The Roller method for particle size determination gives significantly more information than the Blaine method because it analyzes the particle size distribution. This method, however, is much more time consuming and its accuracy in the below-10- μ -diameter range is uncertain because of inherent limitations. This is unfortunate because it is precisely this range which makes the biggest contribution to specific surface. Originally the manufacturer submitted only one value, the median particle size, for each sample as analyzed by the Roller method.

At this point it was impossible to make an intelligent or valid comparison between NRL and the manufacturer's results because of a lack of a common basis. Specific surface cannot be meaningfully calculated from median size and likewise mean diameter cannot be calculated from median diameter. All that can be made is a gross conclusion that a higher specific surface should reasonably be expected to have a smaller median. As pointed out earlier, such was not the case. The resulting random scatter is shown in Fig. 3. Particle diameters when plotted against specific surface on this log-log basis should form a straight line. In order to gain a more thorough insight into the relationships in the manufacturer's results, the complete Roller analyses data were requested.

These additional data were plotted on size-distribution curves as shown in Figs. 4, 5, and 6. The lack of points in the region below 5 μ , except for sample "d," was a handicap and required undesirable extrapolations. The specific surface values were calculated from these extrapolated curves.

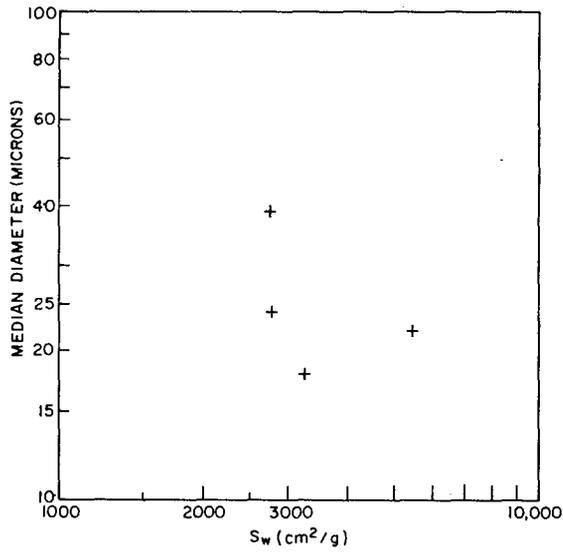


Fig. 3 - Relationship of Roller method median particle to Blaine method specific surface

Fig. 4 - Particle size distribution sample "a" - Roller method

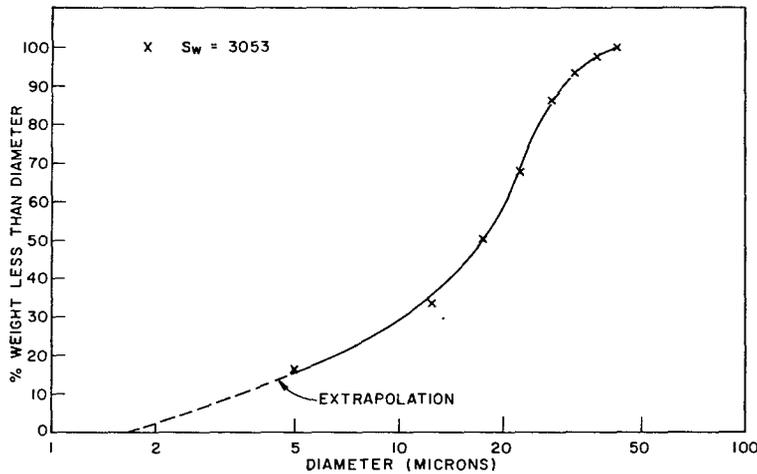
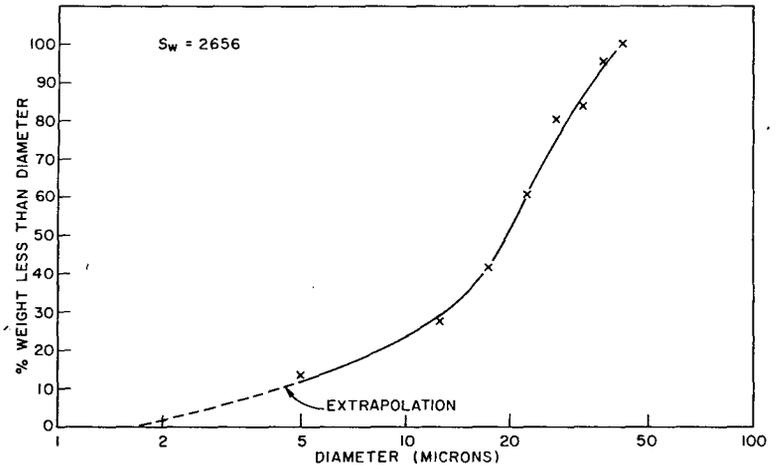


Fig. 5 - Particle size distribution sample "c" - Roller method

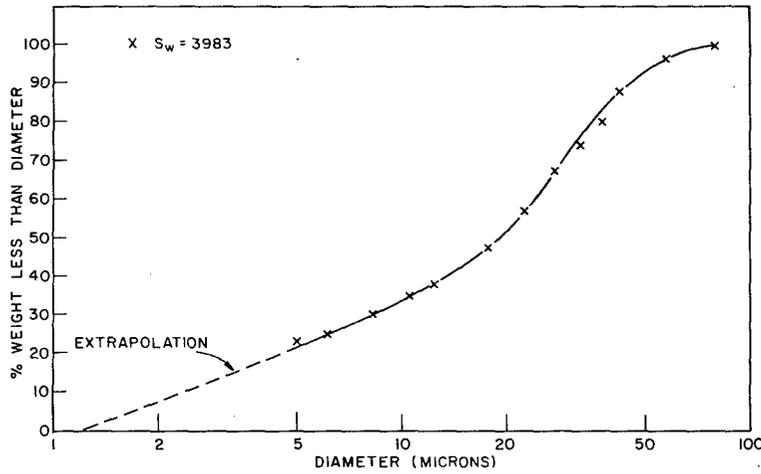


Fig. 6 - Particle size distribution sample "d" - Roller method

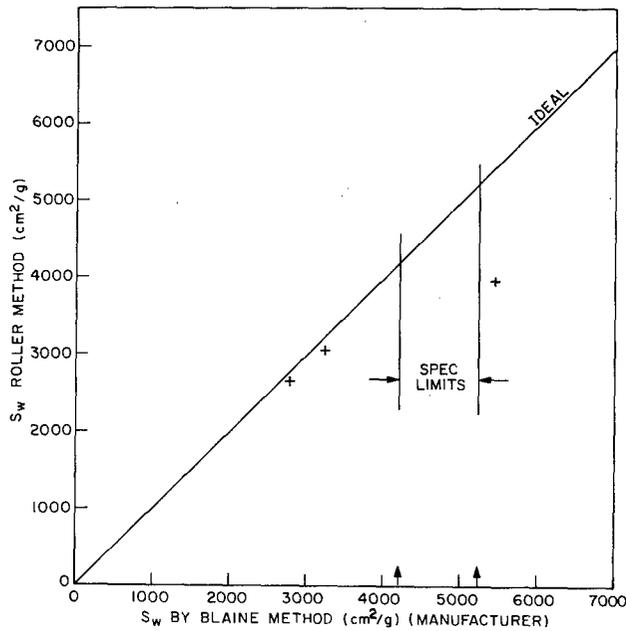


Fig. 7 - Relationship of specific surface by Blaine and Roller methods

By graphically integrating the curves of Figs. 4, 5, and 6, the specific surface for each sample was calculated (3). This enabled a meaningful comparison among the Roller results the Blaine results, and the "ideal" line (see Fig. 7). (All points would fall on the "ideal" line if a correlating constant between methods were always unity.) Immediately a new and distinctive trend becomes evident in contrast to the scatter observed in Fig. 3, and two of the three points available lie very close to the "ideal" line.

The Coulter Counter manufactured by Coulter Electronics, Chicago, Illinois, is a device which determines the number and size of particles suspended in an electrically conductive liquid.* This procedure offered a cross-check on the other techniques and used a completely different principle than either of the others. Figures 8, 9, 10, and 11 are graphs of the particle size analysis data from tests conducted by Coulter Electronics in their service laboratory.

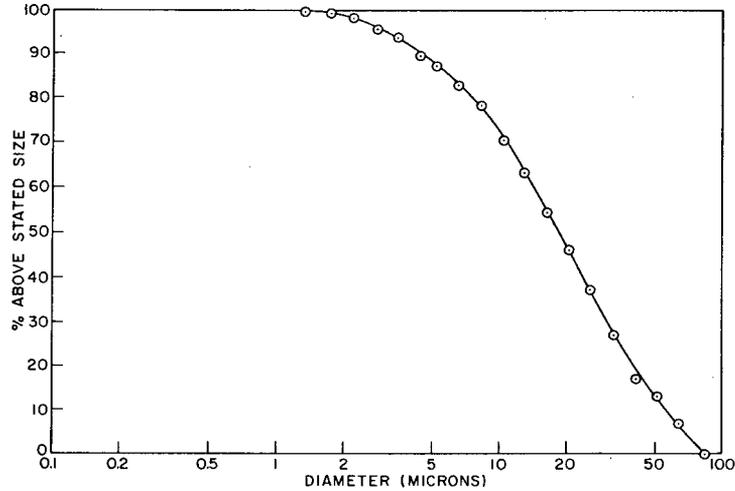


Fig. 8 - Particle size analysis sample "a" - Coulter method

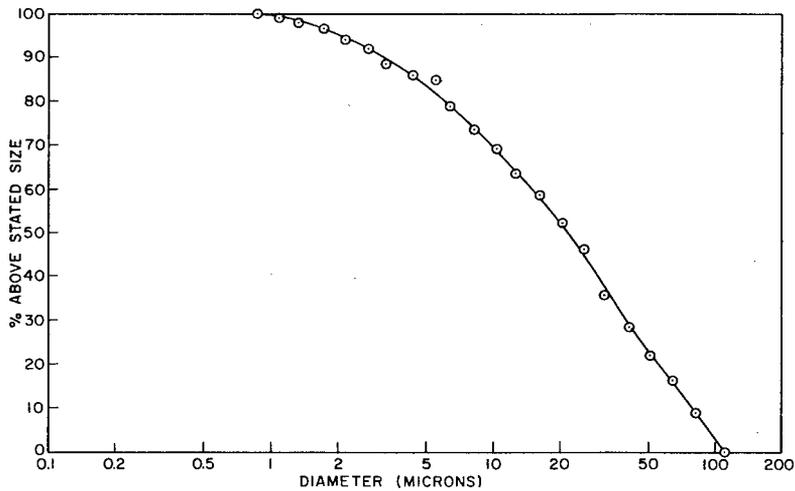


Fig. 9 - Particle size analysis sample "b" - Coulter method

A plot showing the relationship between the Coulter data and the powder manufacturer's data (Roller method) is given in Fig. 12 using the values for both mean and median diameters. It is quite evident that the mean values are better related to the "ideal" line than are the median values.

*See the appendix for a description of the Coulter Counter.

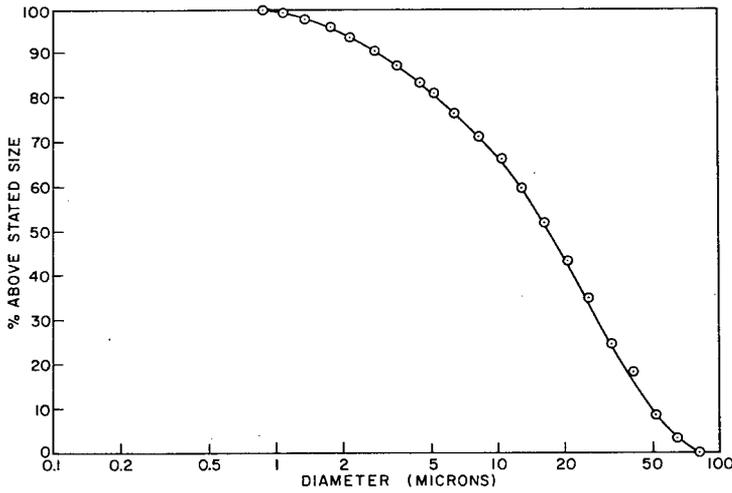


Fig. 10 - Particle size analysis sample "c" - Coulter method

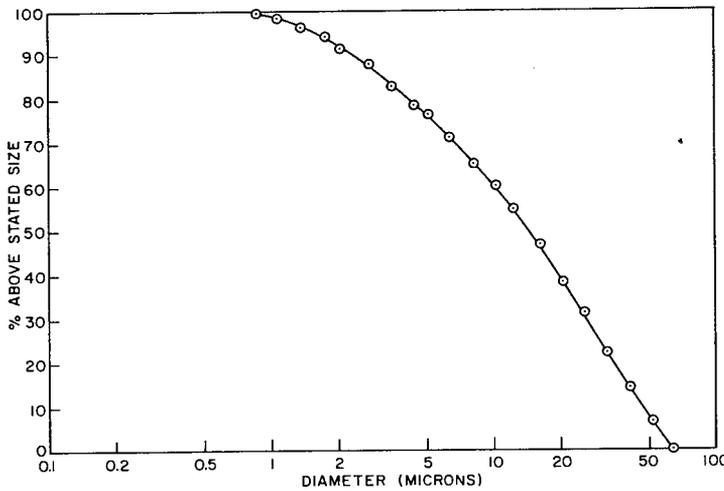


Fig. 11 - Particle size analysis sample "d" - Coulter method

A plot showing the relationship between the manufacturer's Blaine, Roller, and Coulter results and the NRL Blaine results is given in Fig. 13. This plot summarizes all the data from the three sources. In addition, two other Coulter points, one very coarse and one very fine, available from other test work, have been added to extend the range of comparison.

DISCUSSION OF MEASUREMENTS

The Blaine technique for establishing the relative particle size of a powder has advantages because it is modest in apparatus cost and rapid in operation, as compared to other methods like the Roller, Coulter, and Micromerograph. In an earlier study the reproducibility of results with the Blaine method was found to be acceptable, with results

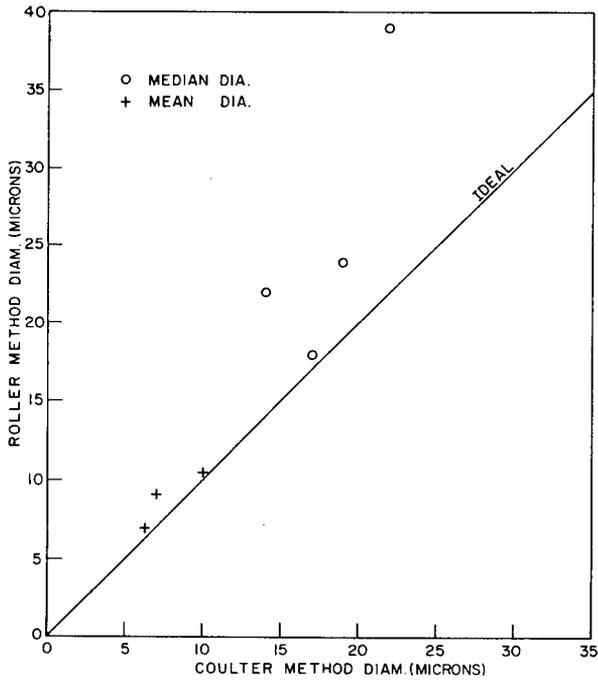


Fig. 12 - Particle size comparisons by Roller and Coulter methods

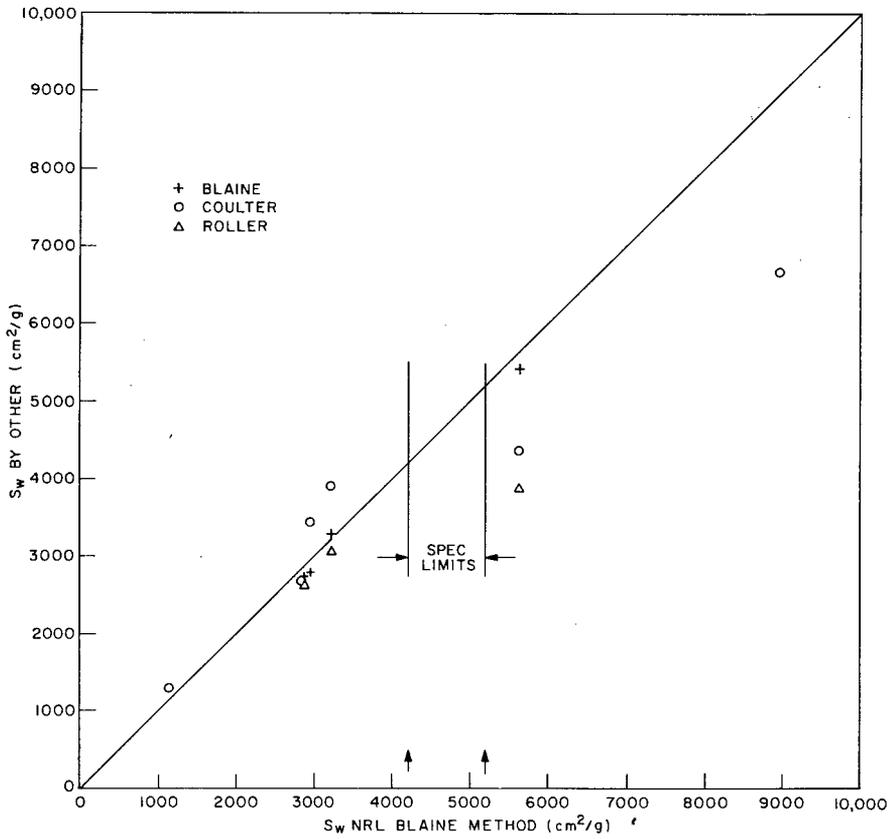


Fig. 13 - Specific surface results compared by methods

within $\pm 6\%$ between different operators and laboratories. Again, Fig. 1 of this report well illustrates this point. If the Blaine procedure meets the level of required precision, the next question concerns the accuracy. Unfortunately, in particle size work there is no absolute standard by which to judge the accuracy of the measurements of irregularly shaped particles having a porous nature. Researchers, therefore, must seek an indirect and roundabout approach.

The first step was to compare the Blaine results with other procedures which might be considered to be the same in principle, permeametry, one such procedure is the Fisher Sub sieve Sizer (ASTM B-330). This comparison was made and the results were very close for dry chemical samples over the entire wide range of 1000 to 10,000 cm^2/g specific surface.

The second step was to compare results with other procedures which might be considered to be more fundamental in principle, such as the Roller Analyzer, which is based on air elutriation. The data generated by this method were used by the manufacturer to establish the median particle diameter, or what they designate as the "average" particle size. It means that 50% by weight of the powder is smaller than this diameter and 50% is larger. The surface area varies as the square of the diameter, and such a single point cannot and does not relate to particle surface area and consequently could not be expected to correlate with Blaine results. The lack of a straight line alignment of the points in Fig. 3 lucidly demonstrates this fact.

By the simple expedience of utilizing all of the information which can be gained from a Roller determination on a powder sample, much more meaningful results are obtained. A comparison of specific surface values from the Roller and Blaine procedures (Fig. 7) shows that the two samples low in specific surface are almost equal. At some region above a specific surface of about 3500 cm^2/g the values begin to diverge, with the Blaine method giving high areas and the Roller method giving low areas. The current military specification limits are noted on this figure and it can be seen that they lie in the area where the relationship is non-linear. This makes it impossible to use the two methods interchangeably for specification testing. There is no indication, however, of which method is the most accurate, but the distribution of points in Fig. 7 gives rise to the possibility of constructing a conversion graph to enable converting results from one basis to the other.

The third step was to introduce the Coulter Counter in order to see if it would verify one or the other of the previous methods. Figure 12 illustrates the more linear relationship of mean particle diameter over median diameter and reinforces the previously expressed thought that median diameter is an inadequate descriptive value for specific surface. The plot of Fig. 13 compares the specific surface results of the identical samples as run by NRL with the Blaine method and/or other techniques and laboratories and serves as a summary of the data. Good agreement with the Blaine method is achieved with both the Roller method and Coulter method below 4000 cm^2/g . Above this value the Coulter result is intermediate between the others and probably represents the most accurate picture. This is true because it is generally assumed that the Blaine method results are too high and that the Roller results are too low.

The product resulting from a grinding process normally has a particle size distribution described as log-normal (4). One method of checking the validity of the analysis of a ground material sample or of checking the accuracy of a size analysis is to observe the distribution curve for conformity with log-normal distribution. A log-normal distribution plots as a straight line on log-probability graph paper. In Fig. 14 the curves from a Roller run and a Coulter run have been plotted in order to check their pattern. The Coulter method data form a good straight line, while the Roller method data have a pronounced curvature. This suggests that with the Roller method some fraction of the sample, conceivably the finest, was missing or was being lost in conducting the procedure. This figure also suggests that the Coulter data gave a more accurate picture of the size distribution.

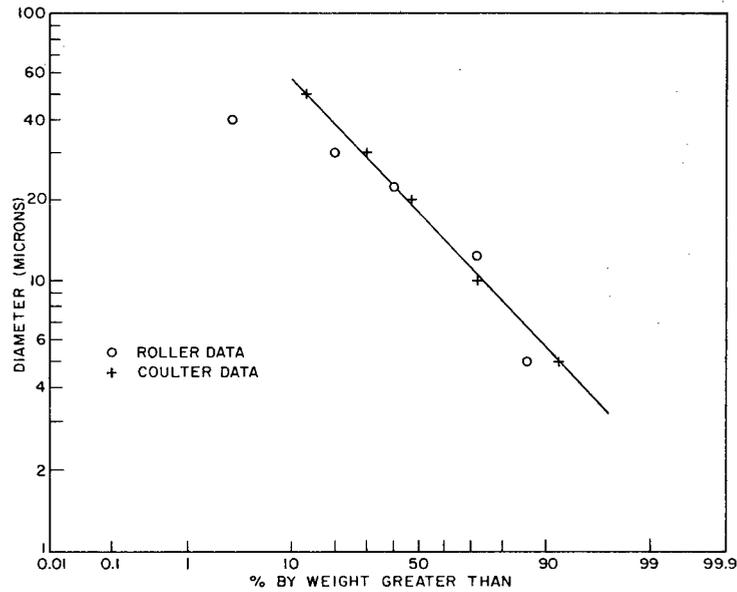


Fig. 14 - Log-probability plot of Roller and Coulter data for sample "a"

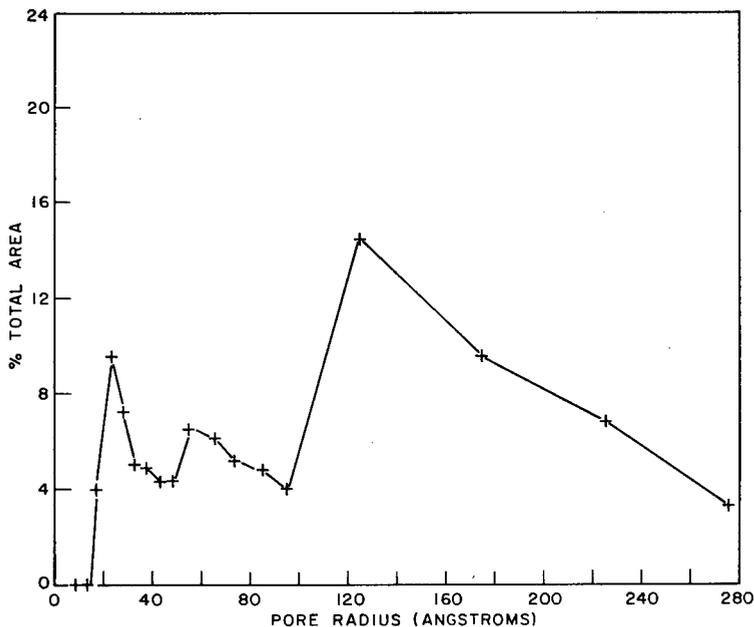


Fig. 15 - Pore size distribution

Total surface area and pore size determinations were made by the American Instrument Company on samples "a" and "d". These were done using nitrogen adsorption-desorption isotherms in the "Adsorptomat" and the Winslow mercury porosimeter. The first method is capable of measuring pore radius distribution in the 7 to 300 Angstrom range, while the second covers the 0.015 to 100- μ -diameter range. Although the dry chemicals are not commonly thought of as being porous, these results showed the B-E-T surface area to be much greater than did the other methods previously cited. Sample "a" with a Blaine value of 2800 cm^2/g had a B-E-T area of 40,000 cm^2/g ; sample "d" with a Blaine value of 5600 cm^2/g had a B-E-T area of 48,000 cm^2/g . Adsorption peaks, as shown in Fig. 15, occurred at pore radii of 23, 58, and 125 Angstroms. Approximately 15% of the total surface area of the powder, 6,000 cm^2 , was in pores of 125A radius.

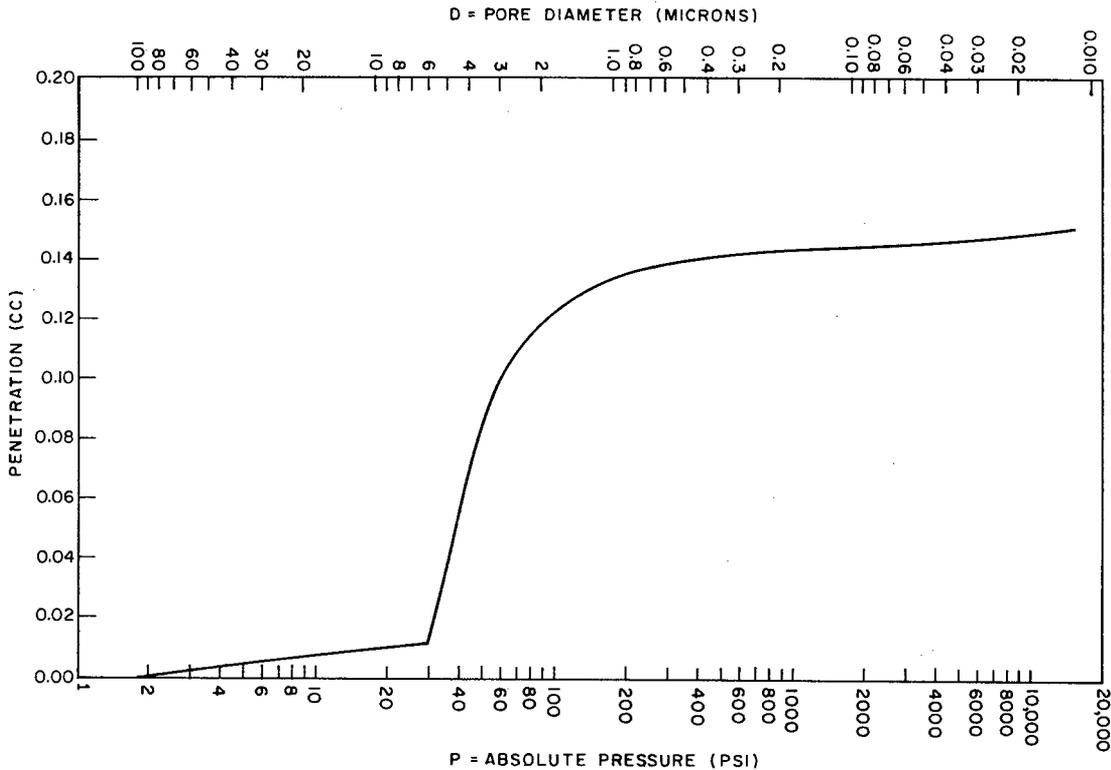


Fig. 16 - Porosity determination

The nitrogen method indicates the sizes of pores or fissures occurring within the KHCO_3 crystals or between crystals which have become attached to one another. These attached crystals would be read as one particle by the Blaine method or other procedures. These surface areas would be available for chemical reactions with molecules, atoms, ions, or radicals smaller than 300A should these play a role in the extinguishing mechanism. On the other hand, if the mechanism is chiefly one of heat transfer to the KHCO_3 particles to promote decomposition to K_2CO_3 , KOH , K^+ , and OH^- , then the normal specific surface area, is probably more indicative of the relative rate of reaction and extinguishment.

The mercury penetration curve resulting from running sample "a" in the Winslow Porosimeter is given in Fig. 16. A very pronounced peak appears at the 4- μ size (Fig. 17). This peak, probably is the point at which the mercury is penetrating the interstitial void spaces between the particles, and is related to particle size and packing arrangement. No differences between samples "a" and "d" could be interpreted as being of significance (5).

A calculation of the mean diameter from the B-E-T area gives a result of 0.7 and 0.58 μ for samples "a" and "d" respectively, or about one-tenth the diameter found by the other methods. This mean "particle diameter" really represents a hypothetical particle whose total surface area would be all on the external surface and not divided between outside surface and internal pores and fissures. The hypothetical particle is called a crystallite in order to differentiate it from the normal particle as seen by the eye.

CONCLUSIONS

The use of median particle size as determined by the Roller analysis and Coulter analysis procedures (and probably others) is misleading and inadequate for the purpose of describing the specific surface of fire fighting dry chemicals.

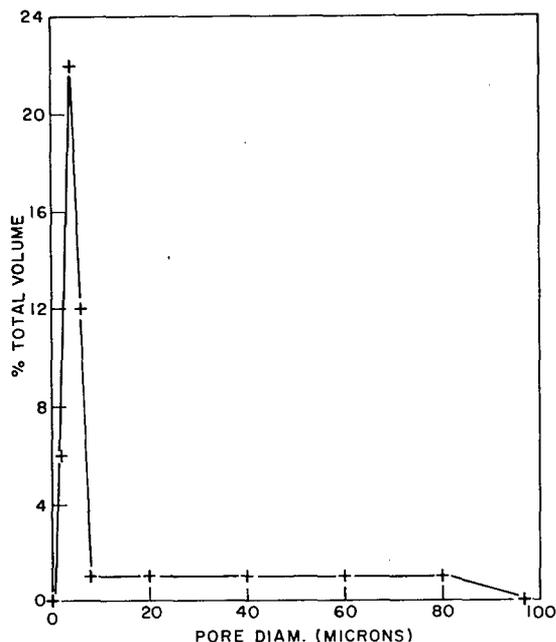


Fig. 17 - Porosity distribution

The mean particle diameter or specific surface is a more accurate significant concept for characterizing a powder's state of subdivision.

The mean particle diameter or specific surface may be determined either from particle size distribution curves or by the Blaine procedure. However, above $4000 \text{ cm}^2/\text{g}$ the relationship among procedures is nonlinear and conversion factors are required to compare values among procedures.

The present Blaine procedure is adequate for military specification purposes until such time as new developmental work can better define the particle size requirements needed for the most efficient fire extinguishing capability.

The total surface area as determined by nitrogen adsorption techniques is a promising tool for evaluating the potential reactivity and fire extinguishing mechanisms of finely divided materials.

RECOMMENDATIONS

It is recommended that the Blaine procedure for specific surface determination be retained in MIL-F-22287.

Although no mention was made in this report of the specification limits for specific surface, it is recommended that the present limits be reviewed with the objective of determining whether the most efficacious material producible is being procured.

It is recommended that studies be undertaken to correlate particle size distribution and particle properties, to fire extinguishing efficiency. The results would lead to better descriptions and better manufacturing control of fire extinguishing dry chemicals.

It is recommended that additional work be done in comparing results of the various particle size measuring techniques to enable the preparation of master conversion charts for relating the techniques.

ACKNOWLEDGMENTS

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APPENDIX

METHODS FOR PARTICLE SIZE AND PORE SIZE DETERMINATION

Throughout the text of this report references are made by name to different methods used in making particle size determinations, specific surface measurements, and pore size distributions. In order to assist readers who may not be familiar with these procedures a brief description of each follows.

Blaine Permeameter - (ASTM C204-55)

This is one of two methods discussed which are based on the resistance offered by a volume of packed material to the flow of a fluid. The stream-line flow of air around the particles gives rise to the flow resistance and therefore, indicates only the external surface areas of the particles and does not include the internal areas contained in the cracks and fissures. A fixed volume of air is forced through the sample bed by a falling column of oil. The rate of fall (thus specific surface) is dependent on the particle size and the porosity of the material by the relationship:

$$s_w = k \sqrt{t}$$

s_w = Specific surface (surface area per unit weight)

k = constant for the apparatus

t = time of flow of fixed air volume

The Blaine apparatus is low in cost and simple to operate.

Fisher Subsieve Sizer - (ASTM Tentative B-330)

This method is also based on permeametry, like the Blaine, and thus "sees" the same things. Here, however, the flow rate of air is measured through the powder bed. This flow is obtained with a controlled pressure differential. An accompanying chart enables the operator to determine the average particle size directly by a graphical solution.

Micromerograph

This is a commercial device manufactured by Franklin Electronics, Inc., Bridgeport, Pa. (Formerly Sharples Research Corp.), which operates on the basic principle of sedimentation and Stokes equation for falling bodies. The rate of fall of a particle through a fluid is related to its density and its "equivalent" diameter. In the Micromerograph many of the basic operating problems have been overcome, resulting in a highly automated instrument. The sample of material is dispersed by a blast of nitrogen at the top of an 8-ft.-long setting column and collected at the bottom on the pan of a recording balance. The resulting time-weight curve can then be translated into a particle size distribution curve using calibration data supplied with the instrument.

Roller Analyzer - (ASTM B293-60)

This is a commercially available device, operating on the principle of elutriation, the reverse of sedimentation as used in the Micromerograph. An upward moving stream of air is used to carry off the finer particles leaving the coarser ones. A metered flow of air carries particles through settling chambers of different diameters and thus different cross-sectional stream velocities. The larger heavier particles settle downward while the lighter ones are carried over, collected by filtration, and weighed. By varying the air flow rates through the various chambers, different size fractions are collected and size distribution data plotted.

Coulter Counter

The Coulter apparatus provides a means for quantitatively sensing the discontinuities which occur in a moving stream carrying insoluble particles. The moving stream is an electrolyte drawn through an orifice supported between two electrodes. Particles suspended in the stream displace electrolyte in proportion to their volume with a corresponding change in electrical current flowing between the electrodes. This results in a pulse in a discriminating circuit. By gradually increasing the sensitivity in this circuit a cumulative count of particles larger than a given size is obtained and thus a complete size distribution curve picture results. Single counts may be made in 5 to 25 seconds.

Adsorptomat

The Adsorptomat is a newly developed instrument for the automatic determination of nitrogen adsorption-desorption isotherms. It is used to find the total surface area of a material including the external area and the internal area resulting from cracks, crevices, and pores in individual particles. Nitrogen is adsorbed on solid surfaces in a monomolecular layer. Thus by knowing the size of the molecule and the amount absorbed, it is possible to calculate the surface area present in a sample. The B-E-T (Brunauer, Emmett, and Teller) theory establishes the relationship between multimolecular thickness and pressure and from only several points at the beginning of the adsorption run of liquid nitrogen at a given temperature the surface area may be calculated. The gas molecules will penetrate pores as small as 7Å in radius and be adsorbed on interior surfaces. Observations of the pressure resulting from adding fixed volumes of gas reveals the pore size characteristics from 7Å to 300Å.

Porosimeter

This device is used for making porosity determinations much in the manner of the Adsorptomat but uses mercury instead of nitrogen to obtain data in the 0.002 to 100μ pore diameter range. Mercury's high surface tension prevents it from wetting the powder, however, it can be forced into the pores by pressure. The greater the pressure used, the smaller the pores that will be filled. Thus, measuring the volume of mercury consumed at each of many pressure levels will generate a pore size distribution curve and reveal more knowledge of the materials' individual particle structure.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Fire Extinguishers Particle Size Determination Specific Surface Area Potassium Bicarbonate Purple-K-Powder Blaine Fineness Test Roller Particle Analyzer Coulter Counter						

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