NATIONAL BUREAU OF STANDARDS REPORT

5424

A METHOD OF MEASURING THE DISPERSIBILITY

OF

LIGHT COLORED POWDERS

by

C. S. McCamy and T. G. Lee

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

A METHOD OF MEASURING THE DISPERSIBILITY OF LIGHT COLORED POWDERS

ABSTRACT

A small sample of powder was dropped in a specified manner from a dispenser at the top of an enclosure. The reflectance of the powder distribution at the bottom of the enclosure was measured with respect to a layer of the same powder in a specified form. One hundred times the reflectance measured in this way was the measure of dispersibility. The dispersibility of powdered sodium bicarbonate was studied as a function of particle size and the dispersibility was found to be a maximum for powder with a mean particle diameter of 15 microns.

1. INTRODUCTION

When a small quantity of a powdered material is thrown into the air, the particles may cling to one another or they may become widely distributed. The characteristic tendency of a powder to become widely distributed in the air is referred to here as the dispersibility of the powder. It is the purpose here to give a definition of this quantity in terms of a specific method of measurement.

The need for a precise definition of dispersibility and a method of measurement arose in the course of a study of the effectiveness of various fine powders as fire extinguishing agents. In a series of fire extinguishment experiments, various powders were roughly graded according to apparent dispersibility (1).* A pinch of powder was dropped, its behavior was observed as it fell through the air, and after several observations the powder was graded A, B, or C. The fire test results showed a general correlation between the effectiveness of the powders and the dispersibility rating. Further investigation of this relationship and the relationship between particle size and dispersibility required a more precise and objective measure of dispersibility.

*Numbers in parentheses refer to references at the end of this report.

The method developed by Puri and Keen (2) was not directly applicable because it involves the preparation of a dispersion of the powder in water and many of the materials of interest were soluble in water. The powders could have been dispersed in a liquid medium in which they were not soluble but the correlation, if any, between the dispersibility in liquid and that in air was unknown. The method developed appeared to measure directly the property of interest in fire research work and required about one-tenth as much time as the method of Puri and Keen.

Andreasen (3) measured dispersibility by dropping one gram of powder about 2.5 meters through still air and noting the weight of powder reaching the bottom, as a function of time. Dispersibility was evaluated on the basis of the percent by weight remaining suspended in the air after 6 seconds. This method makes no distinction between different materials if all of the particles reach the bottom in less than or more than 6 seconds. Even if the material remains partly suspended at 6 seconds, this criterion would not take into account the conditions prevailing at times before or after 6 seconds. Thus, the observed dispersibility index might be the same if 90 percent of the material reached the bottom just after 5 seconds as it would be if 90 percent reached the bottom in 1 second. It would seem that ideally the effect of all parts of the sample should, somehow, be taken into account. This ideal condition is more closely approximated in the present method than in methods previously described.

2. PROCEDURE

The method developed was a quantitative version of the original method of estimating dispersibility by dropping powder and observing its behavior. The procedure consisted of two parts, dropping a measured amount of powder under controlled conditions and measuring the distribution of the powder deposit.

Since various powders were to be compared with respect to their tendency to become spacially distributed, comparisons were made on the basis of equal volumes (0.7 cm^3) of solid crystalline material. Because weighing is simpler and more precise than measuring the volume of irregular solids, the volume of each sample used was measured indirectly by weighing.

The weighed sample of powder was placed in a brass cylinder with an inside diameter of 1/8 in. and a length of 1 1/4 in. The cylinder was open at one end and was equipped at the other end with a close-fitting piston and a thumb screw having 40 threads per inch to advance the piston. A wire 0.0179 in diameter (25 A.W.G.) was placed diametrically across the open end of the cylinder to break up the powder stream. The cylinder was held in a fixed position with its axis and the cross-wire horizontal and the screw was turned by hand. The powder advanced toward the opening until a small amount projected over the edge of the cylinder, then a section of the powder would fall off. The size of the pieces which fell varied over a wide range with any given powder but seemed to be independent of the rate with which the screw was turned from intermittent operation to 15 rpm. This type of behavior served to make the method insensitive to variations in the discharge rate. The rate used was about 15 rpm.

To prevent the influence of extraneous air currents, the powder was dropped within an enclosure. The enclosure was a cylindrical fiberboard drum with a diameter of 17 in. and a height of 32 in., fitted with a thin metal top and bottom. The powder dispenser was placed in a holder at the center of the top of the enclosure. A glass window in the holder permitted observation of the falling powder. The enclosure could be opened to permit the removal of powder after each run. To allow time for the enclosed air to become still, the dropping of powder was not started until one minute after the drum was closed.

The reflectance of the powder distribution relative to a standard surface of the same powder was taken as a measure of dispersibility. The enclosure was designed so that the reflectance measurements could be made on the powder where it fell. After the powder was dropped and one minute was allowed for the last of the powder to reach the bottom, the powder dispenser was replaced by a photovoltaic cell, Weston type M-856. The photocell was mounted in one end of a cylinder 1 7/32 in. in diameter and 5 1/4 in. long painted flat black inside and a 3/8 in. aperture was placed at the other end to limit the field of view. The mirror filled the field of view of the photocell and reflected only the black interior of the enclosure and the image of the photocell. For the reflectance measurements, the powder distribution was illuminated through two Lucite windows of 2-in. diameter located on opposite sides of the enclosure with centers 4 in. above the mirror surface. Light was provided at each window by a 350 watt photoflood

lamp operated at constant voltage. The positions of the lamps were adjusted slightly when necessary to provide the same illumination from one test to the next. For this purpose, the illumination was measured on a matte-white enameled metal disk 4 in. in diameter placed at the center of the mirror. The output of the photocell was measured with a Leeds and Northrup Microvoltmeter. The response was practically linear in the range used. When the response to the dispersed powder distribution had been obtained, the powder was removed, the mirror was cleaned, and the response to a standard distribution of the same kind of powder was obtained. The standard distribution was a uniform layer of the powder 1/4 in. thick in a circular pan with a diameter of 3 3/8 in. placed at the center of the mirror. One hundred times the ratio of the response with the dispersed powder distribution to the response with the standard distribution was designated the "dispersibility" of the powder.

3. RESULTS

Most of the measurements have been on commercially prepared sodium bicarbonate in the form known to fire protection engineers as "dry chemical" and on the separate fractions of this material obtained by sieving or elutriation. For this material, some correlation has been found between mean particle diameter as determined by an air-permeability technique (Fisher Sub Sieve Sizer) and the dispersibility. The dispersibility was a maximum for powder with a mean particle diameter of about 15 microns. Powders having about half that particle diameter or about twice that particle diameter had a dispersibility only about half as great. Andreasen found a similar correlation for a ceramic powder, although different particle diameters were involved and the definition of dispersibility was different. These results and the values of dispersibility of several other kinds of powder are given in Table 1. In the table, the notation "-400" means the material passed a 400 mesh sieve (ASTM Standard Specification for Sieves for Testing Purposes, E11-39) and the notation "+400" means the material was retained on a 400 mesh sieve. All particle diameters were obtained by the air-permeability technique. The reported dispersibility is the mean of three to five determinations except in one case where only one determination was made. The mean deviations are given in all cases but this one. All determinations were made at a temperature of 70 to 72° F. All of the measurements were made at a relative humidity between 50 and 60 percent except in the case of the material designated "Silica gel No. 2", which was measured at a relative humidity of about 15

percent. This material was in every other respect the same as that designated "Silica gel No. 1". The dispersibility was greater in the drier air. The lowest dispersibility measured in this first series was 24.9 for starch and the highest in this series was 79.8 for the fraction of a commercial dry chemical passing a 400 mesh sieve.

4. DISCUSSION

When the development of a method of measuring dispersibility was undertaken, the view was held that dispersibility implied a "spreading out" and it was the objective to devise a measure of "how much powder fell how far from the center" when dropped in a specified manner. In the first procedure employed, a long narrow glass plate was placed at the bottom of the enclosure to catch powder. The distribution of powder with respect to the distance from the center was obtained by measuring the transmittance of the powder deposit on the plate along one diameter of the distribution. For the purpose of analyzing the data, and with some logical justification, the distribution was considered as a Gaussian error function and the probable error of the distribution, so considered, was adopted as the measure of dispersibility. The method distinguished between powders of high and low dispersibility and consistently ranked materials of high dispersibility but the results were not consistently reproducible with powders of low dispersibility. In the case of powders of low dispersibility, agglomerations of considerable size sometimes fell quite a distance off center without disintegrating. With a bright light placed in the enclosure, it was possible to observe the powder falling from the dispenser to the plate and it was found that agglomerations in the form of thin plates or wedges reached considerable distances from the center by gliding through the air. This observation brought out the fact that materials may disperse in more ways than one and that the mere spreading out of material in any manner whatsoever does not in itself constitute dispersion in the sense intended here. The "error function" method rated materials which dispersed as aggregations as more dispersible than was desirable since it gave a measure of "how much powder fell how far from the center". It was to eliminate this interpretation of the dispersal of aggregations that a change in the method was desirable.

The basis for the method adopted was essentially the theory of hiding power and opacity of paints. Finely divided matter can cover a greater area than the same weight of material in large lumps. It is a corollary that light-colored powders on a dark background reflect more light if they are well dispersed than if they are distributed in large agglomerations.

The mirror was chosen for a background because with the mirror facing the unilluminated black surfaces on the interior of the enclosure it reflected less light than any black material illuminated by the lamps. Furthermore, between experiments the glass mirror could be cleansed of all powder more easily than most black surfaces.

The measurement of reflectance with respect to a thick layer of a certain area of the same powder rather than a specified standard white surface cancelled the effect of the color of the powder. The standard layer chosen reflected about one-fourth more light to the photocell than the powder distribution resulting from dropping the most dispersible powders tested. Though comparison of the distribution to a uniform layer covering the whole field might have been a less arbitrary procedure, keeping the two values of luminous flux in the same range minimized photometric errors. In either case the procedure would be arbitrary because the amount of powder dropped and the height of drop were arbitrarily selected.

All of the powders of interest in the work for which this method was developed were near white in color and the method was particularly designed for light-colored materials. It would appear that if it were necessary to work with dark materials, the mirror could be replaced by a clear or translucent plate and the transmittance rather than reflectance of the deposit could be measured by a suitable photometric system. Such a system might be applied equally well to light and dark powders and the correlation between dispersibilities determined by transmittance and reflectance could be obtained for the intercomparison of data.

The method described is rapid and easy to use. It does not require a very large amount of powder nor does it involve very elaborate or delicate apparatus. It has sufficient precision for the intended purpose.

5. REFERENCES

(1) McCamy, C. S., Shoub, H.; Lee, T. G.; Fire extinguishment by means of dry powder. Sixth International Symposium on Combustion, August 1956 (proceedings in publication)

(2) Puri, A. N., and Keen, Bernard A.; The dispersion of soil in water under various conditions. J. Agr. Sci., 15, 147-161 (1925)

(3) Andreasen, A. K. M, Hofman-Bang, N., and Rasmussen, N. H; Uber das Staubungsvermogen der Stoffe, Kolloid-Zeits, 86, 70 (1939)

Table 1

Material	Fraction	Particle Diameter* microns	Dispersibility average	No. of determinations	Mean deviation of dispersibility
Dry Chemical No. 1	-400 (elutriated)	7.3	49.2	3	1.6
Dry Chemical No. 2	unsieved	11.5	58.2	3	1.3
Dry Chemical No. 1	-400	12.7	79.8	5	2.3
Dry Chemical No. 1	unsieved	17.3	70.8	5	1.2
Dry Chemical No. 1	-400 (residue after elutriation)	19.0	78.6	5	1.6
Dry Chemical No. 1	-325 + 400	24.5	68.6	4	2.5
Dry Chemical No. 1	-270 + 325	28.0	59.1	5	1.4
Dry Chemical No. 1	-230 + 270	30.6	44.8	5	1.4
Dry Chemical No. 1	-200 + 230	33.6	29.7	1	---
Sodium Borate	unsieved	5.0	33.8	3	0.3
Silica gel No. 1	unsieved	4.0	33.3	3	0.7
Silica gel No. 2	unsieved	4.0	39.1	2	1.4
Cornstarch	unsieved	11.2	24.9	5	0.6

*As measured by air permeability method

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