

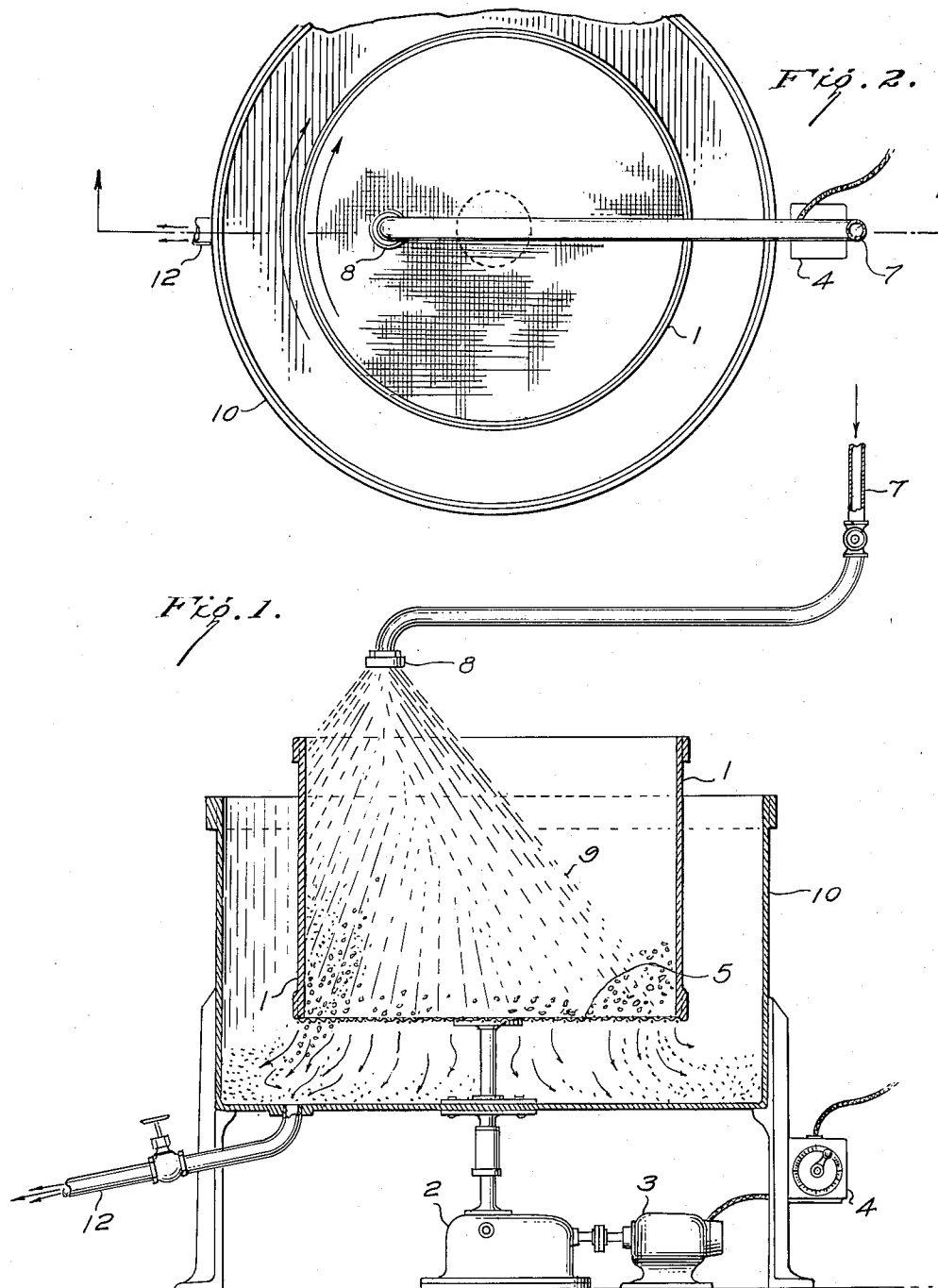
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GRADING PROCESS AND APPARATUS

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1

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GRADING PROCESS AND APPARATUS

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This invention relates to a wet process for grading particles and to an apparatus useful for carrying out the process. More particularly, the invention relates to a process and an apparatus therefore for testing samples of particulate material for the percentage of fines or coarse particles in the material by impinging a sheet of grading liquid on particles of the test material supported on a rotating sieve.

The process of this invention is generally designated a wet grading method in the art. The word "grading" connotes the determination of the particle size distribution in a given sample of particulate material. As a rule, the value sought is the percent of particles in a sample which are too large to pass through a given size of sieve opening. Manufacturers and users of pigments, for example, are often interested in the value mentioned because the color value or tintorial power of a pigment depends to a large extent on the size of the pigment particles. Over a practical range of sizes, the finer the pigment particles, the higher the tintorial power. There has been a need in the art for a process and apparatus for determining with reproducible results the percent of retention on a sieve of oversized pigment particles. A standard test recognized by both the pigment manufacturer and the user would eliminate much disagreement between the buyer and seller of the commodity when the analysts of the two parties find different "percent retentions" for the same pigment sample by using the unreliable manual tests of the prior art.

Previously, particles have been graded by both dry and wet methods. The dry method commonly consists of placing a known quantity of the material to be tested on the uppermost and coarsest sieve of a nest of sieves and agitating the sieves either manually or mechanically. The material retained on each of the successively finer sieves is weighed and its percentage of the original sample calculated. Dry grading methods are generally slow, subject to error due to spillage and loss of material as fly-dust, and as a rule, give non-reproducible results because of the human errors introduced in the method. The human errors are the variation in the amount of agitation from one analyst to the next or even between different experiments by the same analyst, and equally serious, the determination of the end point of the operation. It has been noted that analysts employed by pigment manufacturers tend to be more diligent in shaking and brushing the material on the sieves and are also apt to work for a longer period of time on a given sample. This, of course, is due to the analyst's desire to find as low a percent retention as possible for his employer's product. The analyst employed by the pigment user or buyer has no particular desire to find a low percent retention and is, therefore, less diligent than the manufacturer's analyst and is almost certain to get a different result. The dry method is also objectionable since the fly-dust settles on laboratory equipment creating a considerable nuisance in many cases.

2

Wet methods of grading particles have been found to be somewhat more satisfactory than the dry methods described above. In the usual wet grading process, a weighed sample is brushed under a stream of water from a tap or other source through a nest of sieves as in the dry grading process. The mechanical losses due to spillage and loss of material through fly-dust are obviated. However, the human errors discussed above are also introduced here, so the wet grading method has not proven very satisfactory. Also, reproducible values are difficult to obtain by this method because the percent retention varies somewhat with the velocity of the grading liquid impinging on the particles, the type of spray or orifice employed, the duration and type of brushing used and the method of wetting out or dispersing the material tested. As in the dry method, the most serious causes of error are the variation in diligence between analysts and the judgment of the end point of the operation. The wet methods of the prior art have the additional disadvantage that the grading liquids used, while chosen for their inertness, frequently do have some slight solvent action for the material to be graded. This fact, plus the possibility of attrition of the particles by long brushing or agitation on the sieve, makes it desirable to keep the time of operation of the process to a minimum to prevent inaccurate results.

According to the present invention, I have found that the wet grading of particles may be materially improved and the disadvantages of the prior art methods eliminated by placing the particles to be graded on a horizontal screen, rotating the screen at a predetermined rate, and directing a sheet of grading liquid at a predetermined velocity rate substantially along a radius of the screen.

Referring to the accompanying drawing which illustrates a suitable apparatus for carrying out the present invention, Fig. 1 is a side elevation, partly in section, of a preferred embodiment, and Fig. 2 is a top plan view thereof.

Reference numeral 1 represents a suitable sieve provided with a horizontal circular screen 5 of suitable mesh. The sieve is rotated by variable speed drive 2 and motor 3 controlled by rheostat 4. The grading liquid enters through conduit 7 and is discharged from orifice 8 which may be of any suitable design so as to produce a sheet of liquid 9 which impinges on the particulate material on the screen along a radius of the screen at an angle of from 30° to 90° from the horizontal. The grading liquid passes through the screen into receptacle chamber 10 and may be drawn off through conduit 12 and recycled or otherwise disposed of. Screen 5 may be of any suitable mesh depending upon the size of the particulate material being graded. Three mesh screens up to 400 mesh screens have been satisfactorily used thus grading particles which vary from 6350 microns to as low as 37 microns in size.

In carrying out the present invention a weighed sample of a particulate material to be graded is pasted with a suitable wetting agent to insure dispersion and a measured amount of liquid, usually water, is added to the slurry and stirred with a high speed stirrer at a definite rate for a definite period of time. The slurry is transferred to the sieve 1. The motor 3 of the apparatus is started and the speed of revolution of the sieve is regulated to a definite number of revolutions per minute by means of the rheostat 4. A speed of 10 to 20 revolutions per minute has been found to be most suitable. The grading liquid may be any suitable liquid which is a non-solvent for and non-reactive with the material to be tested. Water is usually employed since most pigments and materials tested are practically insoluble in water. The grading liquid is supplied through conduit 7 at a definite pre-

determined rate of flow, usually about 4 liters per minute, but the rate may vary widely depending on the size of the apparatus and the size of the orifice used. The sheet of water is directed from orifice 8 so as to fall along a radius of the screen, the angle between the screen and sheet of water being from 30° to 90°.

It has been found that the velocity of the sheet of grading liquid impinging on the particles is important for satisfactory operation of the device. The velocity should be sufficient to properly agitate the particles on the screen and break up possible aggregates of particles, but should be low enough to prevent spattering of the particles out

the analyst. The operation of the apparatus is largely automatic requiring no fatiguing manual shaking by the operator. The working time required is greatly decreased over the time required to test a sample in the prior art. Most tests are completed in 20 minutes as against an hour or more previously required. The chief advantage however of this invention is that results may be obtained which are not affected by the bias of the analyst and may be reproduced from analyst to analyst and from laboratory to laboratory.

The following data are typical of those obtained by the apparatus of this invention.

Product	R. P. M. of Sieve	Velocity of Stream Flow, in. per sec.	Volume Water, liters per minute	Time, Minutes	Percent Retention on Sieve No. 325 ¹
1. Lake made with dichlorobenzidine-diazo and aceto-acet-o-toluidine.	10	70	3.2	20	11.98 12.20 11.72 11.70 12.02 17.20 15.10
2. β -chloro anthraquinone	10	82	3.75	20	15.14 16.80 16.44
					Percent Retention on Sieve No. 200 ²
3. Meta-Nitro-p-toluidine	10	90	4.0	20	0.61 0.72 0.70
4. Meta-Nitro-p-toluidine	20	90	4.0	20	0.63 0.69 0.75

¹ U. S. Standard Sieve No. 325—National Bureau of Standards Letter Circular LC 534

² U. S. Standard Sieve No. 200 (Ibid).

of the sieve or any possibility of causing significant attrition of the particles by abrading them against the screen. It has been found that a velocity of from about 10 inches/second to 200 inches/second produces satisfactory results, but that optimum results are secured when the velocity is about 90 inches/second. The apparatus is allowed to run for a definite period of time and then stopped. The operation may be stopped automatically, if desired, by means of a suitable automatic timer. It has been found that sieving the particulate material for 20 minutes is about the optimum time for most materials. This time of operation may be varied widely so long as the time is constant for a series of tests in which replicate values of percentage retention are desired. It is desirable to keep the test time to a minimum, of course, because of the desirability of obtaining rapid results and the possibility of attrition and solvent action on the sample in a prolonged test. The residue on the screen is dried and weighed in the conventional manner and its percent retention calculated.

The process of this invention and the apparatus described permit an analyst to obtain reproducible results on a series of samples of a pigment or on the same sample in a series of tests. It is possible for the manufacturer and user of the pigment to agree on a standard test to be used in evaluating the pigments sold and used. It is merely necessary to stipulate the speed of rotation of the sieve, the velocity of the sheet of grading liquid, the time of operation, and such minor details as the grading liquid used and the manner of pasting the material to be tested, in order to establish a standard test for any material.

The process and apparatus of this invention eliminate all of the troublesome human errors of the prior art grading methods. Each particle on the rotating screen is contacted the same number of times by the sheet of grading liquid thus insuring uniformity of action. Mechanical losses of material are reduced to nil because there is no possibility for loss through fly-dust or spillage. The degree and kind of agitation is standardized from test to test and does not depend on the diligence or peculiarities of

The table below is given to show how results differed between analysts previously and how substantial agreement of results is possible now by employing this invention.

Comparison of percent retention by the manual wet grading method and the method of this invention

[The particles graded were the lake made with dichloro-benzidine-diazo and aceto-acet-o-toluidine. Sieve No. 325 was used in both methods, and the retention is in percent of the original sample.]

Analyst	Laboratory	Percent Retention by Manual Method	Percent Retention by Method of This Invention
No. 1	A	10.36 10.71 10.18	11.47 12.00 12.07 12.00 12.28 11.48 11.70
No. 2	A	10.62 10.22 9.80	12.02 11.96 12.20 11.72
No. 3	A	12.15 13.91 13.90	13.90
No. 4	B	13.90	13.90
No. 5	B	12.10	12.10
No. 6	B	15.70	15.70
No. 7	C	9.93 7.68	9.93 7.68
No. 8	C	10.58 11.94	10.58 11.94

¹ A velocity of stream flow of 90 inches/second was used.

It is to be noted that the results obtained by the new process and apparatus do not necessarily agree with the results obtained on the same sample by the old manual methods. The important feature is that the results be reproducible. The manual wet method used in the tests above is that of A. S. T. M. Designation D-185-45; Standard Methods of Test for Coarse Particles in Pigments, Pastes, and Paints, modified to wash the material

5

6

on the sieve for 30 minutes. It is to be noted that all of the data obtained by analyst #7 in the table above was only one-half as large as that obtained by analyst #6. Also, the data obtained by analysts #1, 2, 7 and 8 are lower than those obtained by the method of this invention while the data obtained by analysts 3, 4 and 6 are higher.

If desired, multiple sheets of liquid directed to impinge along several radii of the rotating screen might be employed in this invention. It is sometimes desirable to employ a nest of sieves with sources of grading fluid above each sieve. In such a case several size gradations are evaluated simultaneously.

I claim:

1. A method of screen testing for particle size analysis whereby the percentage of particles in a given sample of larger than a specified size is determined comprising: weighing a sample of the material to be tested, pasting said sample with a wetting agent in an aqueous phase to form thereby a uniform dispersion of the sample as a slurry, charging said weighed sample to a rotatable cylindrical drum having a horizontal screen at the bottom thereof and contiguous upstanding sides forming a sieve, rotating said sieve at a predetermined rate, directing a sheet of liquid at a predetermined rate of flow and pressure and for a predetermined time against the screen at an angle of from 30° to 90° from the horizontal to

wash the smaller particles through the screen, whereby the sample particles are classified according to size, the velocity of the sheet of liquid and the rate of rotation of the cylinder being insufficient to cause any appreciable attrition of the sample particles on the screen, drying the residue on the screen, weighing said residue, and calculating the percentage of said residue based on the original sample weight.

2. A process according to claim 1 in which the velocity of the liquid impinging on the screen is between about 10 inches per second and about 200 inches per second.

3. An apparatus for screen testing for particle size analysis, whereby the percentage of particles in a given sample of larger than a specified size is determined comprising: a circular screen, contiguous upstanding sides attached to said screen, forming a circular sieve, means to rotate said sieve at a uniform controllable rate, and means to supply a grading liquid at a predetermined rate of flow and at a velocity between 10 and 200 inches per second as a sheet along a radius of the sieve, for a predetermined time, thereby retaining within the sieve all particles too coarse to pass the sieve.

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