

Jan. 8, 1946.

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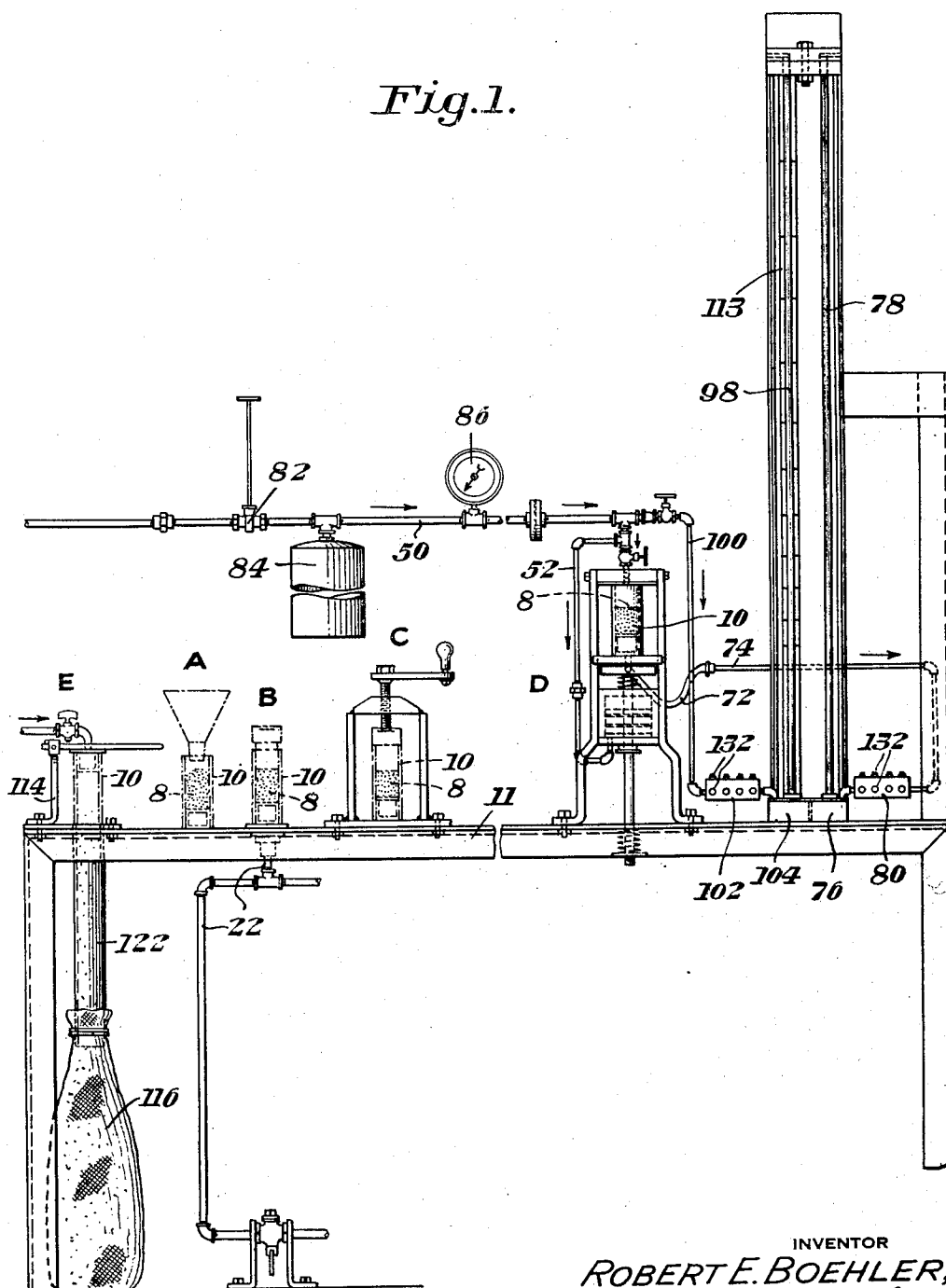
2,392,636

METHOD AND APPARATUS FOR DETERMINING TOTAL SURFACE IN GRANULAR BEDS

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4 Sheets-Sheet 1

Fig. 1.



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4 Sheets-Sheet 2

Fig. 2.

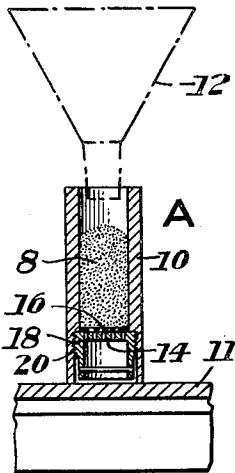


Fig. 3.

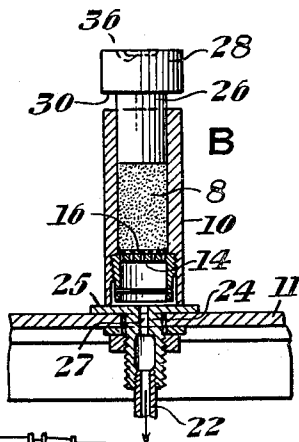


Fig. 4.

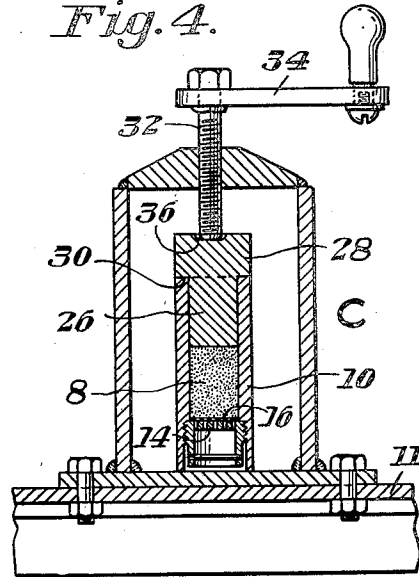


Fig. 5.

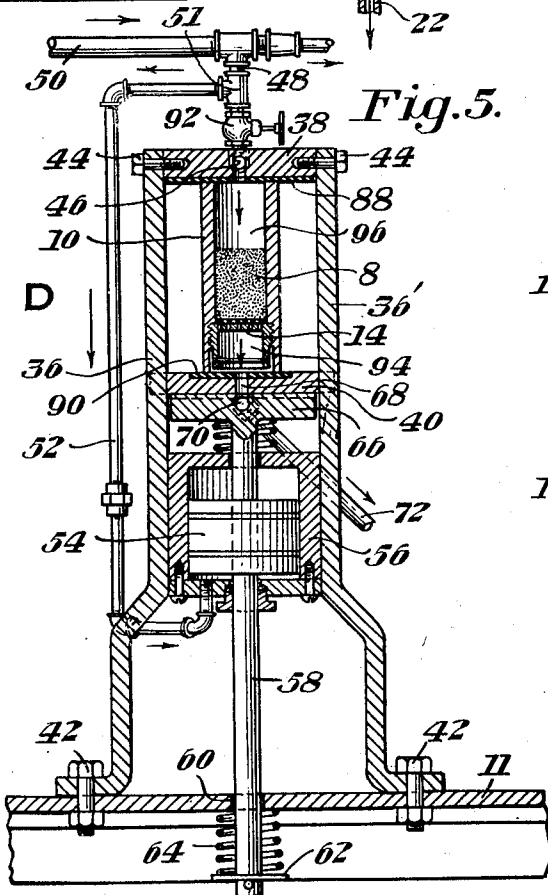
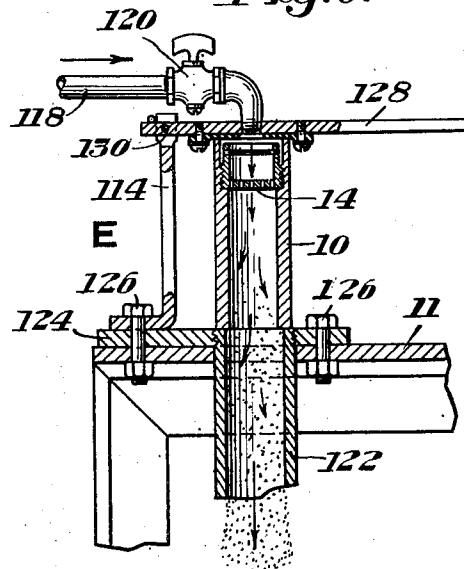


Fig. 6.



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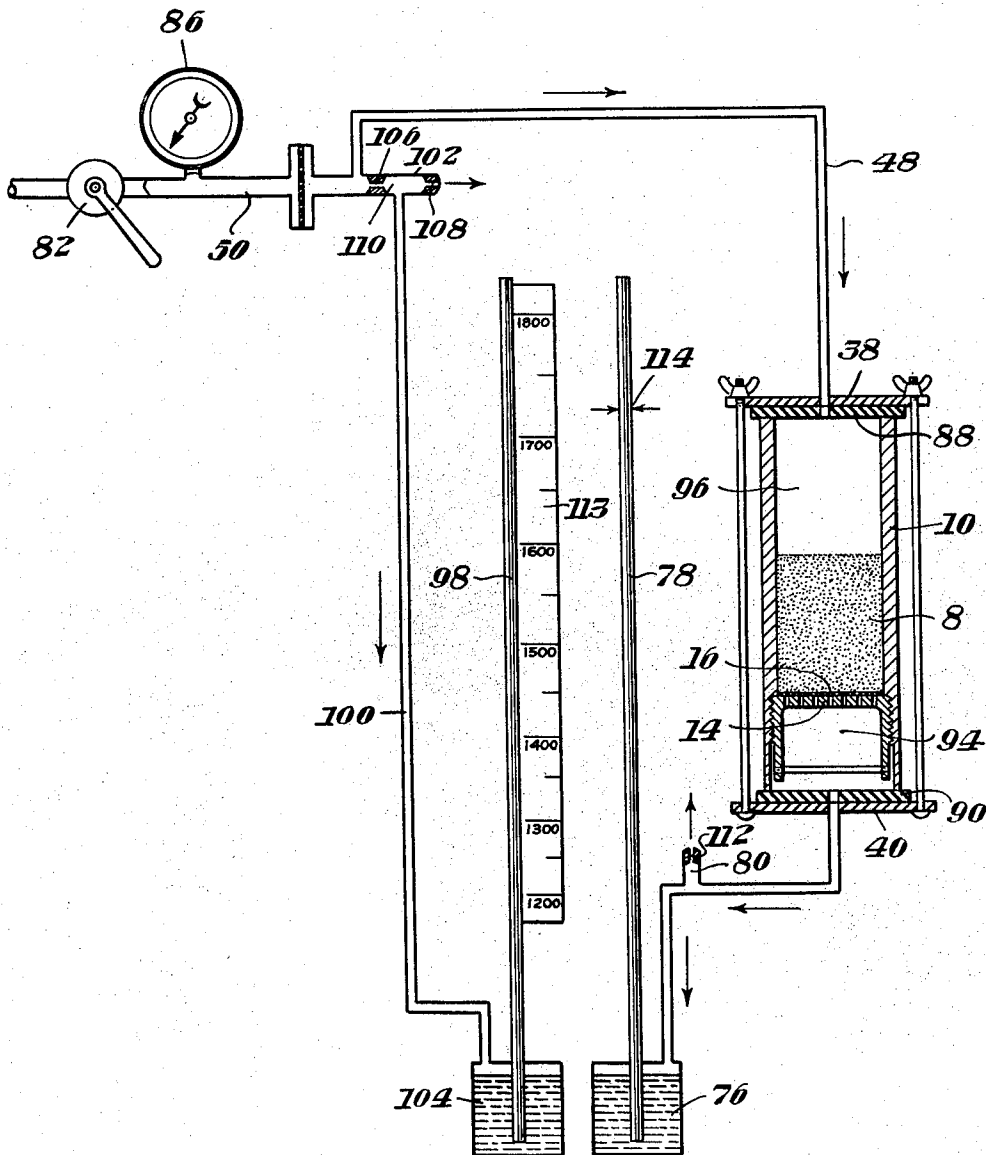
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4 Sheets-Sheet 3

Fig. 7.



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4 Sheets-Sheet 4

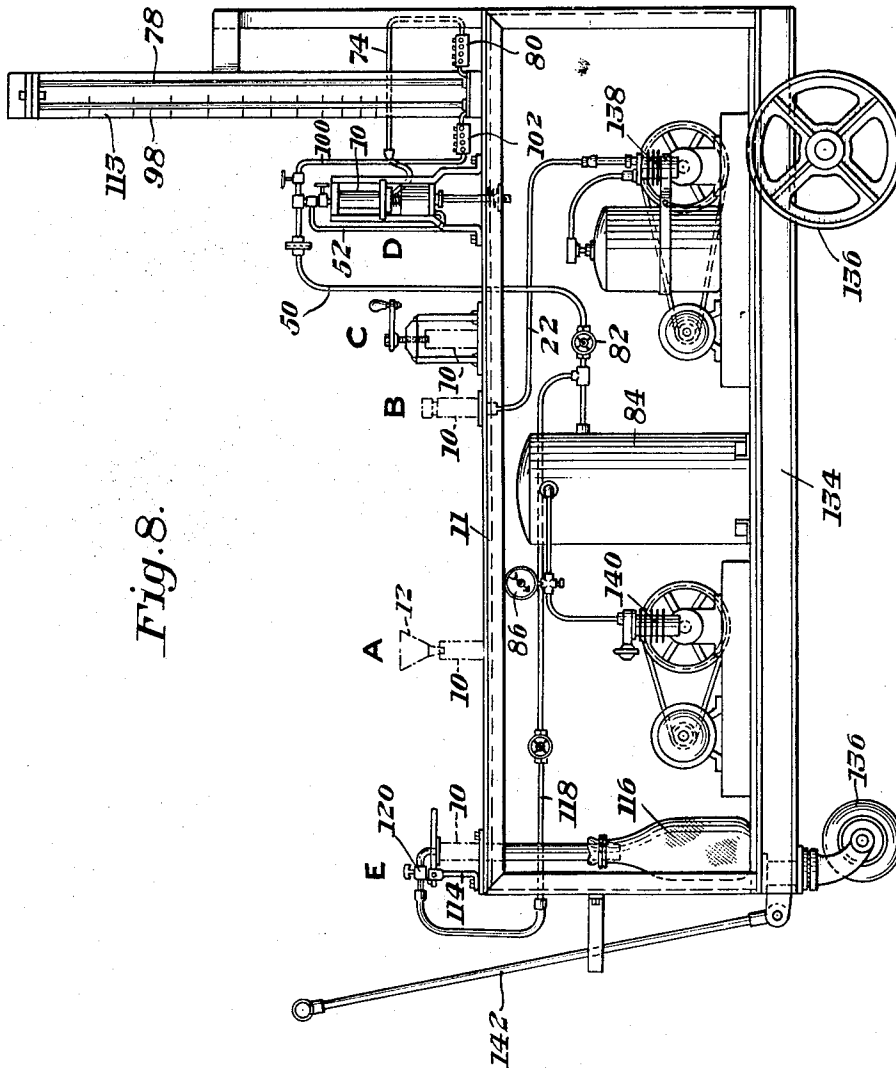


Fig. 8.

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UNITED STATES PATENT OFFICE

2,392,636

METHOD AND APPARATUS FOR DETERMINING TOTAL SURFACE IN GRANULAR BEDS

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Application December 21, 1942, Serial No. 469,709

6 Claims. (Cl. 73—38)

In the production of powdered materials such as cement, importance may be attached to the degree of fineness of grinding of the material, since in the case of cement there is a direct relation between the fineness of the cement particles and the setting properties of the cement. Thus, it is generally accepted that the coarser particles in cement are practically inert, and it is only the extremely fine powder that possesses adhesive or cementing qualities. The more finely cement is pulverized, all other conditions being the same, the more sand it will carry and produce a mortar of a given strength.

In the early stages of hardening of cement, only the finer particles have any effect, as water is slow in reaching the interior of the larger particles, thereby delaying the hydraulic action. Also, the finer particles more easily cover the sand grains making mortar much stronger and allowing the case of a large percentage of sand. Also seasoning can take place more easily with finely ground cement; because of this, fine cement is less liable to unsoundness.

In view of the importance of the fineness of the grinding, it becomes a necessity to make frequent tests on the material during the grinding process in order to effect requisite adjustments of the mill in the event that through some cause the mill is not performing satisfactorily. In view of the fact that the checks should be made frequently during the grinding, it is necessary that they be done in an expeditious manner as the grinding proceeds.

In accordance with the present invention there are provided a new process and apparatus for determining through surface determinations, the fineness of materials such as cement, or for that matter, virtually any finely comminuted substance, cement being the illustrative embodiment of the invention which is illustrated and described herein.

A further object of the invention is to provide a method and apparatus which combines speed with sufficient ruggedness of mechanical construction to enable the equipment to be installed adjacent to grinding machinery to take advantage of test results for regulating the grinding operation.

A still further object of the invention is to provide test equipment which is suitable for use by a person such as a mill operative and not necessarily by a trained laboratory technician and which eliminates the use of delicate laboratory apparatus.

It may be mentioned in this connection, that a still further object of the invention may be stated to be the provision of equipment on which there may be taken direct readings in terms of surface, thereby avoiding the necessity for calculations.

In accordance with the present invention, there is measured the flow of a fluid (specifically air) through a standard bed of the powder to be tested, and expressing the results in terms of surface.

This is accomplished on the basis of the Carman equation for determining surface by the permeability of liquids through beds of granules. When using air as the permeating medium, and its density is expressed in terms relative to the density of fluids, the fineness of the granules also, is assumed to be relative.

P. C. Carman (Journal of the Society of Chemical Industry, vol. 57, 1938, page 225; vol. 58, 1939, page 227) derived the principles involved, and the present invention embraces the method, and inseparably therefrom, the calculations that are involved, together with a compact apparatus for carrying out the method as stated above, the invention being predicated upon the relations expressed by the aforesaid Carman equation for determining surface by the permeability of liquids through beds of granules. When air is used as the permeating medium and its density is expressed in terms relative to the density of fluids, the fineness also may be assumed to be relative.

In the Carman equation, the variables affecting specific surface are porosity, kinematic viscosity and permeability. The relations may be derived in the following manner:

D'Arcy expressed the flow of fluids through granular

$$1. \quad \frac{Q}{A} = K \frac{h}{L}$$

in which:

Q is the volume of fluid passing through the bed in unit time,

A the area of the bed

L its depth

h the loss of head across the bed

$\frac{h}{L} = i$, the hydraulic gradient

$$u = \frac{Q}{A} = Ki$$

K is a factor related to permeability of the bed.

Kozeny's equation expressing the flow of fluids through granular beds is given as:

$$2. \quad u = \frac{\Delta P g \epsilon^3}{L k n S^2}$$

in which

u is $\frac{Q}{A}$ as above

ΔP , the pressure difference across the bed,
 g , the gravity constant, numerically 980,
 ϵ , the porosity of the bed, or the fraction of the total volume not occupied by solid particles,
 L , the thickness of the bed,
 k , a constant, numerically 5.0,
 S , the surface,
 n , the viscosity of the fluid in poises or dyne-seconds

The porosity ϵ is calculated as follows:

Assume a weighed quantity of powder packed into a chamber whose cross-sectional area is A and, so that the thickness of the powder bed is L , the total volume is AL . Then

$$3. \quad \epsilon = \frac{AL - \frac{W}{d}}{AL}$$

in which, using C. G. S. units since surface is expressed in square centimeters per gram:

W , is the weight of the sample, in grams.

d , its density or specific gravity, or grams per cubic centimeter.

$\frac{W}{d}$ = the volume in cubic centimeters of solid material composing the powder.

ϵ , so determined is a ratio expressed as a decimal, and in the case of cement will usually be found to calculate as from 0.4 to 0.5 of the volume of the bed, as voids among the solid grains.

S , signifies the total surface per unit volume of the bed, and it is necessary to relate surface to unit volume of the powder.

Let S_0 represent the surface per unit volume of the powder, then

$$4. \quad S = (1 - \epsilon) S_0$$

Again, if we represent by S_w the surface per gram of powder:

$$5. \quad S_w = \frac{S_0}{d} = \frac{S}{d(1 - \epsilon)}$$

Accordingly Equation 2 may be rewritten for later discussion:

$$6. \quad \frac{Q}{A} = \frac{\Delta P g \epsilon^3}{k L n (1 - \epsilon)^2 S_0^2}$$

in which Q is to be expressed in cubic centimeters per second and all other factors in C. G. S. units.

Carman demonstrated the validity of this formula with particles such as beads or other small granules whose surfaces could be determined independently by experiments, in which liquids were used.

In a bed through which a liquid percolates under its own head, let:

V be the kinematic viscosity of the liquid expressed as square centimeters per second.

P its density, grams per cubic centimeter.

n its viscosity in poises or grams per centimeter per second.

$$V = \frac{n}{P} \text{ or } n = PV$$

Since ΔP in grams per square centimeter represents the pressure difference across the bed.

$$5. \quad \frac{\Delta P}{P} = h, \text{ the loss in head and}$$

$$\frac{h}{L} = i, \text{ the hydraulic gradient}$$

Substituting in Equation 6

$$10. \quad \frac{Q}{A} = u = Ki = \frac{\Delta P g \epsilon^3}{L P V k (1 - \epsilon)^2 S_0^2}$$

$$Ki = i = \frac{g \epsilon^3}{k V (1 - \epsilon)^2 S_0^2}$$

15 and by cancellation of i , an expression for permeability K is derived by which:

$$K = \frac{g \epsilon^3}{k V (1 - \epsilon)^2 S_0^2}$$

With reference to the foregoing, attention is called to the accompanying drawings, wherein

Fig. 1 represents a side elevation of an apparatus for practicing the method of the present invention.

25 Fig. 2 is a sectional elevation of the sample receiving cylinder, showing a sample of powder introduced therein.

Fig. 3 is a sectional elevation showing the first step of preparing the sample.

30 Fig. 4 is a sectional elevation showing completion of the preparation of the sample.

Fig. 5 is a sectional elevation of the sample mounted in the test frame for testing.

35 Fig. 6 is a sectional elevation showing a method of removing the sample from the cylinder.

Fig. 7 is an enlarged diagrammatic view of the manometer section embracing the principles illustrated in Fig. 1.

40 Fig. 8 is a view similar to Fig. 1 showing the apparatus of Fig. 1 mounted for portability.

Referring more particularly to the drawings, and first Figs. 1 to 5 inclusive, a sample of powder to be tested which has been indicated at 8 is

45 introduced into a cylindrical cup 10, placed at a sample-filling station A on a table 11, through a funnel 12 or other suitable introducing means.

The cup 10 is adapted to sit on table 11 and is provided with a perforated bottom 14, the perforations being covered with a layer or filter

50 paper or fine-meshed cloth for retaining the powder, this being indicated at 16. The bottom 14 preferably is made removable by forming it as a part of a bracket 18, which is threaded into

55 the bottom portion of the sleeve, as is indicated by threads 20.

As is shown in Fig. 3, the table 11 is provided with a suction station B which is immediately adjacent to the sample-filling station. The suction station includes a suction line 22, which

60 opens at the top of the table 11, through a suitably apertured fitting 24, the top of which fitting forms a plate 25 having a hole 27 therethrough, the plate 25 being adapted to receive the cylinder 10 with the hole 27 in communication with

65 the interior of the cylinder for evacuation thereof.

The cylinder 10, containing the powder sample 8, is fitted with a piston plug 26, which is

70 milled so as to have a close sliding fit in the cylinder. The piston plug 26 has an enlarged head 28 which forms an annular shoulder 30 adapted to seal on the end of the cylinder 10 when the powder sample 8 is compressed fully.

75 The section station B is located immediately

adjacent to the sample-filling station A, and the cylinder 10 with the piston plug 26 and sample 8 therein, is placed over hole 27 so that when suction is applied through line 22, entrained air is removed from the powder sample, thus initially reducing the volume of powder and causing the piston plug 26 to follow downwardly until the shoulder 30 almost reaches contact with the top of the cylinder 10.

It is evident that as air leaves the powder, mobility of the solid particles produces flow from the cylinder wall downwardly and towards the center, and all layers from bottom to top represent as nearly uniform packing as can be attained at this stage. The sample so compacted remains as a cylinder slightly less in diameter than its metal container, its upper surface under the piston plug slightly higher than required.

Consequently, in order to finish compacting, the cylinder 10 with its contained partially compressed sample and piston plug is moved to a compression station C where a screw 32 operated by crank lever 34 is caused to press upon the piston head 28 in recess 36 provided therein for this purpose until the shoulder 30 is forced into engagement with the cylinder 10, thereby completely compressing the sample for purposes of this invention.

Before applying pressure from the screw 32, the cylinder of powder is not in contact with the walls of the metal cylinder 10, the powder particles have some freedom of movement under pressure until restrained by the metal wall. The powder appears to have some further degree of compressibility so that, for instance among cement samples varying enough in fineness and specific gravity to affect the bulking tendency, it is possible to compress a standard weight within the length allowed. It is conceivable that, with great pressure, enough packing could be developed as to impair the quantity ϵ in Equation 3 above, so that this quantity, as calculated, would not represent the true porosity of the bed. Adjustment of the weight of the sample and degree of mechanical pressure is a matter for experiment not concerned with the present invention, which provides a means for compacting the sample into an accurately dimensioned cylinder in such condition as to satisfy requirements for this test.

The cylinder 10 with its sample thus compacted, then is removed from the press C and placed in testing frame D.

This frame is fastened to the table 11 immediately adjacent to the press C, and includes a frame having sides 36, 36', a top 38 and a bottom 40. The sides 36, 36' are suitably bolted to the table 11 by bolts 42, and the top 38 also is secured suitably such as by screws 44. The top 40 has a hole 46 communicating with a pipe 48 which joins compressed air line 50. A junction 51 connects pipe 48 with a branch pipe 52 opening under a piston head 54 positioned in a cylinder 56 mounted on the side frame members 36, 36'.

The piston head 54 actuates a piston rod 58 which is shown as extending through a hole 60 suitably provided therefore in table 11. A washer 62 is provided adjacent to the bottom end of piston rod 58, and a cushioning coil spring 64 is retained around the rod 58 between washer 62 and table 11.

The piston rod 58 carries a plate 66 at its upper end, which bears against and is secured to the bottom 40, which is slidably mounted in the sides 36, 36', which sides form guides for the movement of the bottom 40. The bottom 40, and

plate 66, are provided with connecting holes 68, 70, to the latter one of which is connected a tube 72 which in turn is connected with a pipe 74 for supplying air under pressure to the well 76 of an oil manometer 78 through a needle escape valve 80.

Pipe 50 is connected to a source of compressed air which is controlled by a valve 82, there being a pressure chamber 84 in the line 50 and also a pressure gage 86.

It will be understood that the powder sample 8 is of standard weight, and when properly compressed as described above herein, the pressure which is required to force air through such standard bed is the value which, in accordance with the present invention, is to be measured.

Consequently, when the cylinder 10 is placed in the test frame and the valve 82 is opened, air flows through the pipes 50 and 52 beneath the piston head 54 to lift the bottom 40 against the cylinder 10, thereby tightly holding the cylinder 10 between the bottom 40 and the top 38. The tight engagements between the cylinder 10 and the top and bottom respectively, are maintained by a washer 88 in the top 38 and a washer 90 in the bottom 40. A valve 92, which desirably is a two-way valve, preferably may be provided in the pipe 48 to direct the flow of air through pipe 52 to lift the piston head 54 to hold the cylinder in the frame properly clamped in place.

Then when the valve 92 is opened, air is admitted to the sample 8, and penetrates the sample causing pressure to build up in the chamber 94 beneath the sample until oil in the manometer 78 rises to its maximum height under available pressure, excess pressure escaping to the outer atmosphere through valve 80, the pressure also rising very substantially in the space 96 above the sample, this pressure rising to as much as 50 lbs. per square inch. In order to measure this pressure, a mercury manometer 98 is connected to pipe 50 through pipe 100, and a relief valve 102 is provided between the air supply line 50 and the mercury well 104 at the manometer 98. The relief valve 102 includes orifices 106, 108, (Fig. 7) a chamber 110 being provided between the orifices 106, 108, thus avoiding an inconveniently long manometer tube for the manometer 98. As high pressure air flows through the sample, some of the air escapes through the orifices 106, 108, and the pressure is reduced in the chamber 110 sufficiently to permit using a mercury manometer 98 of convenient length.

The pressure in chamber 110 bears a relation to the pressure above the sample of powder 8 that can be determined by calibration throughout a range of pressures. Therefore, if this relation is known, the pressure above the sample can be calculated from the height of the mercury column.

Using a standard weight of powder sample, the pressure required to force air through the sample against the standard back pressure through the orifice 112 of valve 80, varies and is reflected in manometer 98 by the mercury rising to a greater or less height. In practice, it is desirable to calibrate this height of mercury in terms of surface as determined on a Wagner turbidimeter by testing standard powder samples, such as, for example, cement samples, in the Wagner instrument, and then constructing a scale 113 the major divisions of which are numbered in terms of square centimeters surface per gram of powder.

It will be seen therefore, that in accordance with the present invention, high pressure air is used as the sample-penetrating fluid, and oil in

manometer 78 is forced to rise to a fixed point, such as that represented by 114 (Fig. 7). This provides a back pressure on the sample, which is the same for all samples tested. The quantity of air flowing is limited by orifice 112 of valve 80, and by the combination of a fixed back pressure and standard orifice, the object is attained of forcing a volume of air through the sample, which is the same in all cases, if measured at the pressure indicated by manometer 78, only one variable remains to be measured, namely, the height of the mercury column in manometer 98, which, by calibration against samples of known surface, is converted directly into surface values.

From the foregoing, it will be seen that the invention involves the following steps, after the sample has been weighed and poured into the sample-container:

1. Compacting the sample by suction and mechanical pressure in the apparatus provided therefor.

2. Inserting the sample in its container into the airflow apparatus.

3. Admitting compressed air until the oil column in manometer 78 rises to the point 114.

4. Manually adjusting further admission of compressed air until with manometer 78 steady at point 114, the mercury in the manometer 98 rises to some point and stops. Then, after observing that a steady state has been reached.

5. Reading the surface valve on scale 113.

6. Removing the sample container 10 from the air-flow apparatus by closing valve 82 to allow piston head 54 to drop, thereby releasing the container 10, inverting the container and ejecting the sample.

This is carried out at station E on table 11, which conveniently is located as shown. The container 10 is held in inverted position by clamp 114 and the tested sample is ejected therefrom into waste bag 116 by a jet of compressed air from compressed air line 118, controlled by valve 120, the bag 116 being attached to the discharge end of waste pipe 122, which is threadedly mounted in plate 124 bolted by bolts 126 of the table 11.

Clamp 114 is comprised of an upright standard to which arm 128 is pivoted at 130 for vertical movement, and is adapted to be held manually against the sample container as the air from pipe 118 ejects the tested sample as is indicated in Fig. 6.

It is obvious further, that the apparatus of the present invention employs the principle of air flow through the sample, and that surface values may be calculated by means of Carman's equation instead of by calibration against standard samples.

In this connection, Equation 6 above may be repeated for reference:

$$6. \quad \frac{Q}{A} = \frac{\Delta P g \epsilon^3}{k L n (1 - \epsilon)^2 S_o^2}$$

Solving for S_o^2 —this equation becomes

$$7. \quad S_o^2 = \frac{\Delta P g A \epsilon^2}{Q n (1 - \epsilon)^2 k l}$$

Now, knowing the ratio between the height of mercury in manometer 112 and the pressure above the sample, the pressure can be calculated in grams per square centimeter. Likewise, the back pressure in grams per square centimeter can be calculated from the height and the density of the oil in manometer 78. The difference between these is ΔP in Equations 6 and 7 above.

By measuring the volume issuing at orifice 112, the volume flowing the sample under the back pressure can be determined in cubic centimeters and become Q in the above Equations 6 and 7, which then can be written:

$$8. \quad S_o^2 = \frac{\Delta P}{Q n} \cdot \frac{\epsilon^2}{(1 - \epsilon)^2} \cdot \frac{g A}{k L}$$

Examining the pressure-volume-viscosity relation in air flow, it is evident that as compressed air enters the upper surface of the cement powder sample and passes downwardly, the pressure decreases, due to the work expended in overcoming the resistance to flow. If the temperature remains constant by heat transfer from the surroundings, the air volume must increase so that through the sample both pressure and volume are changing.

At the surface of the bed, let the initial high pressure be P , and let the volume entering the surface be V cubic centimeters per second. At any level below the surface, where the pressure has become reduced to P' , the volume of air passing in cubic centimeters per second must have increased to V' to maintain the constancy of the product of pressure and volume such that

$$9. \quad PV = P'V'$$

and so on until at the exit pressure, the volume is Q centimeters per second.

It is evident that at the steady state of flow, as Q cubic centimeters of air leave the sample, an equivalent amount of air must enter each second at the upper surface. Let P be the density of air leaving, then $QP = M$, the mass in grams per second. If P_v represents the density of the volume V of air entering at the surface at pressure P , then

$$10. \quad QP = VP_v = M$$

Since $n = PV$, and $QP = M$, then the relation that holds throughout the bed is $Qn = MV$ or the mass times the kinematic viscosity. Hence the Equations 7 and 8 for surface may as well be written:

$$11. \quad S_o^2 = \frac{\Delta P}{MV} \cdot \frac{\epsilon^2}{(1 - \epsilon)^2} \cdot \frac{g A}{k L}$$

Therefore, apart from the use of the apparatus of the present invention as an industrial instrument by calibrating against standard samples, it is possible to calculate surface values independently, precision depending on accurately determining the rate of flow at orifice 112 and the viscosity of air at the pressure prevailing there and the temperature.

The present invention, by its construction and operation, requires establishing a constant rate of flow, the same for all samples. Since manometer 78 performs the function of establishing a standard back pressure on the sample, only one variable is to be determined, which variable is the height of the mercury in the manometer 98, which is the feature that permits direct reading of surface values on a scale.

Valves 80 and 102 are made adjustable, the amount of air flowing through the respective orifices being closely controllable by means of valve adjustments 132.

While the invention has been specifically illustrated and described in connection with the testing of cement, it will be understood that it is adapted for the testing of powders generally, and that its use is not intended to be limited to the specific embodiments as herein illustrated and described.

The apparatus of the present invention is constructed so as to be a compact, self-contained unit, which can be readily portable, so that it may be moved from place to place if desired. The respective elements are mounted on the stand or table so as to be in easy arm's length of an operator making the test, and the apparatus is sufficiently sturdy to enable it to be placed adjacent to a grinding mill without adverse effects due to the vibrations of the mill.

It will be apparent further, that while it is preferred to utilize the step of evacuating entrained air from the sample, as described herein, this step may be omitted and the powder sample compacted by mechanical pressure alone to form the standard sample bed.

Fig. 8 shows the apparatus of Fig. 1 mounted on a carrying truck whereby the apparatus is rendered freely portable. The same reference numbers are applied to the same parts as are shown in Fig. 1. Truck 134, together with its supports, are mounted on wheels 136, a suction pump 138 being mounted on the truck to provide required suction for evacuating the powder in cylinder 10 and a compressor 140 also being provided for the testing and cleaning lines. Handle 142 is provided for pulling the equipment around the plant to any station of desired use.

It will be understood, of course, that while air is illustrated and described herein as the penetrating fluid, other fluids may be used in a similar manner, so long as they are inert to the particular powder being tested. Thus a mobile oil may be used in the case of cement, or water in the case of powders not reacted upon with water. Consequently air is to be requested as illustrative only of a fluid that is found to be satisfactory in practice, easily available, and easily controlled.

What is claimed is:

1. The method of determining fineness of a powder through determining total surface of a bed thereof, which comprises introducing a standard weight of sample of the powder into a container, evacuating entrained air from the sample while partially compressing the same, completing compression of the sample into a bed of predetermined dimensions by applying mechanical pressure thereto, forcing compressed air through the said bed, measuring pressure differentials through the bed, and directly converting the said measurements into terms of surface.

2. The method of determining fineness of a powder by determining total surface of a bed thereof which comprises introducing a standard weight of sample of the powder into a container, evacuating entrained air from the sample while initially partly compacting the same, mechanically compressing the evacuated and initially compacted sample to complete compacting thereof into a bed of predetermined dimensions and density, forcing a test fluid through the said bed, measuring pressure differentials through the bed, and directly converting the pressure differential measurements into terms of surface.

3. The method of determining fineness of a cement by determining total surface of a bed

thereof which comprises introducing a standard weight of finely comminuted cement into a receptacle having a perforated bottom, applying the receptacle containing the sample to a suction line, exhausting entrained air from the sample by suction applied thereto by the suction line, thereby partially compacting the sample, completing compacting the sample into a standard bed by applying mechanical pressure thereto, forcing compressed air through the resulting bed, establishing a constant pressure at the outlet side of the bed, measuring the said constant pressure, while simultaneously but separately measuring the pressure on the intake side of the bed, and converting this measurement into terms of surface.

4. In testing a sample of powder for total surface by measuring pressures produced by forcing a penetrating fluid through a standard bed of the sample, the improvements which consist in preparing the sample for testing by weighing a standard amount of sample in a container, evacuating entrained air from the sample while effecting a partial compression of the powder, and then applying mechanical pressure to complete the compression of the powder sample into a predetermined bed.

5. Apparatus for testing a sample powder for total surface thereof, which comprises a holder for securing in testing position a container holding a sample of powder to be tested, the holder including a top, a movable bottom adapted to receive the container and to lift the container against the top and for clamping the said container, means in the top of the holder for supplying fluid under pressure to the sample, a lifting piston for the bottom of the holder, means for supplying fluid under pressure to the piston for actuating the piston thereby moving the bottom of the holder between clamping and releasing positions, and means for connecting the said sample to pressure indicating means adapted to measure pressure differentials through the sample.

6. A testing unit for testing fineness of powdered cement, which comprises the combination with a stand, of testing instrumentalities mounted on the stand, the said stand with its instrumentalities mounted thereon being adapted to be positioned in a cement plant adjacent to a grinding mill, the said instrumentalities including holding means for receiving a prepared bed of the cement of predetermined weight, dimensions, and density, the said holder means having an intake side and an outlet side disposed relatively to the said prepared bed for enabling passing of compressed air through the bed, means on the holder for passing compressed air through the said bed, separate pressure measuring devices, means for connecting the intake and outlet sides of the holder to the said pressure measuring devices, the said devices indicating pressure differentials through the bed, and means for converting the indicated pressure differentials into surface values.

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