

[72] Inventors **Archibald James Morcom;**
Kenneth John Burr; Terence Wilfrid
Webb, Cornwall, England

[21] Appl. No. **749,507**

[22] Filed **Aug. 1, 1968**

[45] Patented **Mar. 30, 1971**

[73] Assignee **English Clays Lovering Pochin & Company**
Limited
St. Austell, Cornwall, England

[32] Priority **Aug. 7, 1967**

[33] **Great Britain**

[31] **36220/67**

[56]

References Cited

UNITED STATES PATENTS

2,981,630	4/1961	Rowland	210/54X
3,009,388	11/1961	Polanyi	73/61.4
3,391,597	7/1968	Gropper	250/218X

Primary Examiner—James W. Lawrence

Assistant Examiner—T. N. Grigsby

Attorney—Larson, Taylor and Hinds

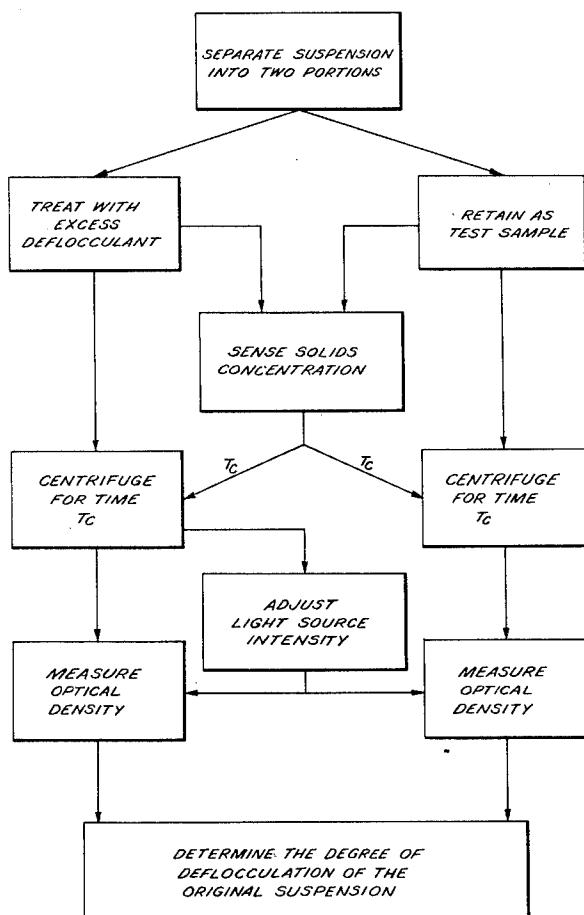
[54] **APPARATUS AND METHOD FOR MEASURING**
THE DEGREE OF DEFLOCCULATION OF A
SUSPENSION OF FINE PARTICLES
9 Claims, 4 Drawing Figs.

[52] U.S. Cl. **356/36,**
73/61.4, 73/432, 250/218, 356/72

[51] Int. Cl. **G01n 1/00,**
G01n 15/04, G01n 21/00

[50] Field of Search **356/36, 72;**
250/218; 73/61.4, 432 (PS), (Inquired); 210/54;
233/19

ABSTRACT: An apparatus and process for measuring and controlling the degree of deflocculation of a suspension of fine particles. The apparatus includes a centrifuge having two separate compartments into one of which there is introduced a portion of the suspension under examination and into the other of which there is introduced a further portion of the suspension under examination which has been treated with excess deflocculant so as to completely deflocculate said further portion. The compartments of the centrifuge are each provided with a window whereby light can be passed through the portions of the suspension thereunder. Photocells are employed to measure the optical densities, and thus the solids concentrations, of the parts of the suspension in the compartments through which the light is passed and this enables the degree of deflocculation of the suspension to be ascertained.



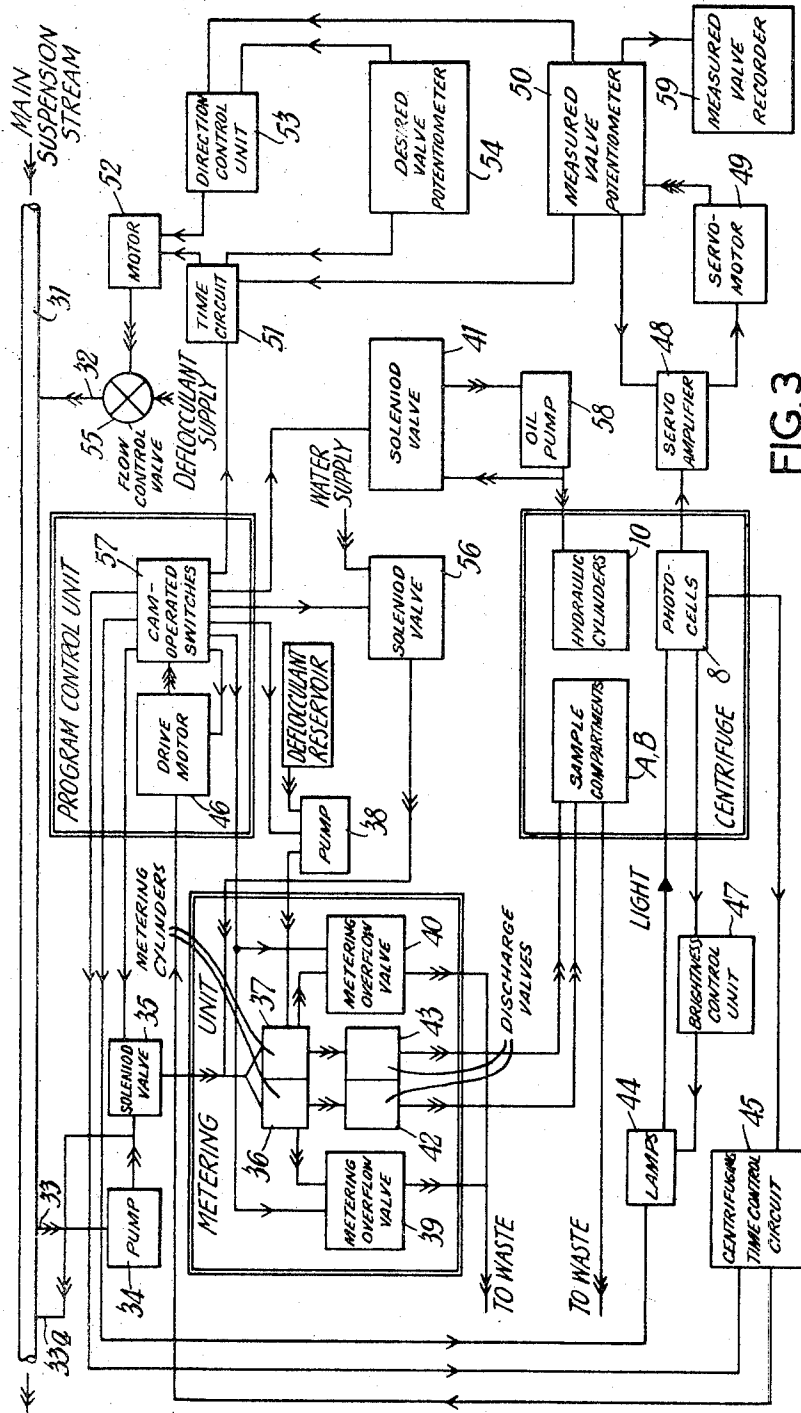
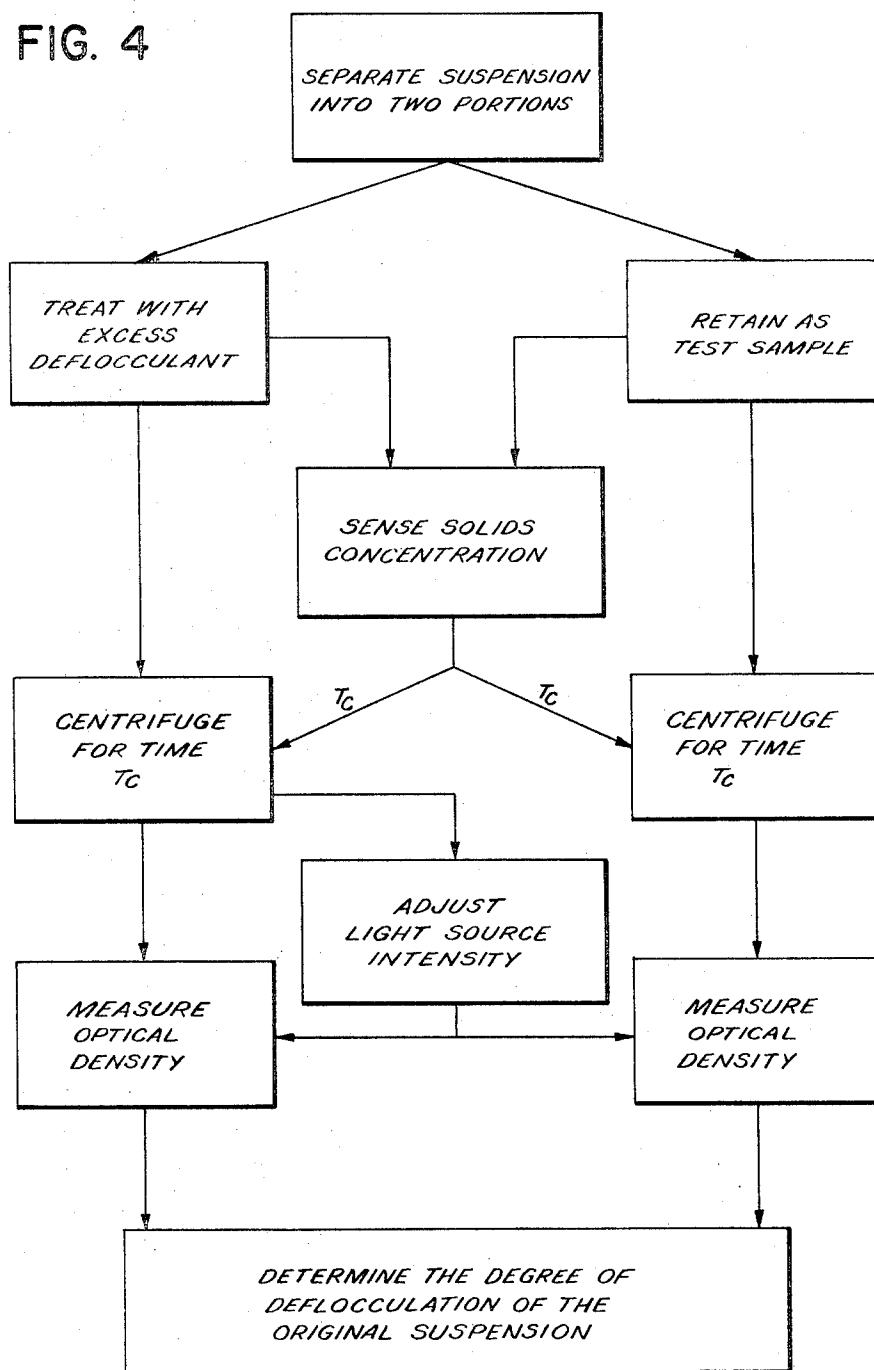


FIG. 4



APPARATUS AND METHOD FOR MEASURING THE DEGREE OF DEFLOCCULATION OF A SUSPENSION OF FINE PARTICLES

BACKGROUND OF THE INVENTION

This invention relates to an apparatus and process for measuring, and optionally controlling, the degree of deflocculation of a suspension of fine particles, for example a clay slip.

Whenever a suspension of fine particles, i.e. particles substantially smaller than 100 microns, is subjected to a particle size classification process, it is common practice to deflocculate the suspension prior to the particle size classification process. Deflocculation is a physico-chemical process in which, by the absorption of suitably charged ions, the fine particles become equally charged giving rise to repulsive forces between the particles, thus overcoming the attractive Van der Waals forces and allowing the particles to move independently and not as a cohesive floc of particles of variable size. Commonly used deflocculants are sodium silicate, sodium polyphosphates and sodium salts of poly-(acrylic acid). The usual method of controlling the addition of deflocculant is by control of pH. This has the disadvantage that the pH at which a suspension of fine particles reaches a given degree of deflocculation is not an invariable but depends upon several factors such as the type of mineral and its previous chemical treatment. It is inevitable, therefore, that with the pH control set to cover all eventualities, the addition of deflocculant will often be greater than that really required. Also, the method is not suitable for use with deflocculants which do not cause a change in pH.

It is an object of the present invention to provide a process and an apparatus which will enable the degree of deflocculation of a suspension of fine particles to be ascertained.

SUMMARY OF THE INVENTION

It is found that when a suspension of fine particles sediments for a given time, the solids concentration of the suspension at a point a little way below the surface of the suspension is dependent on the degree of deflocculation. By the term "solids concentration" there is meant herein the weight of solids in a given volume of the suspension. The greater the degree of deflocculation, i.e. the more deflocculated the suspension, the greater will be the number of finer particles present, which settle more slowly and therefore the higher the finer particle concentration in the upper levels.

If a quantity of a suspension of fine particles, of which it is desired to determine the degree of deflocculation, is divided into two portions and an additional quantity of deflocculant is added to one portion sufficient to cause complete deflocculation, then a comparison of the solids concentrations of the two portions of the suspension at identical depths and after identical sedimentation times will give a measure of the degree of deflocculation of the original sample, the other factors, such as initial solids concentration and particle size distribution, having been eliminated.

It is therefore a further object of the present invention to provide a process and an apparatus which will enable there to be carried out rapidly a comparison of the solids concentrations of suspensions of fine particles at identical depths and after identical sedimentation times.

Accordingly, in one aspect the present invention provides an apparatus comprising a centrifuge having two separate compartments each provided with a window, whereby light can be passed through material present in said compartment, and a photocell arranged so as to interrupt the light passing through said window and the material in said compartment and to produce a signal corresponding to the optical density of material present in said compartment at a fixed distance from the axis of rotation of said centrifuge, and means for comparing the signals obtained from each compartment.

According to another aspect of the invention there is provided a process for measuring the degree of deflocculation of a suspension of fine particles which process comprises

separating said suspension into two portions, adding to one of said two portions a quantity of a deflocculant sufficient to completely deflocculate said one portion, introducing the portions of said suspension one into each of two separate compartments of a centrifuge, said two compartments each being provided with a window, whereby light can be passed through the portion of said suspension therein, and each having associated therewith a photocell arranged so as to interrupt the light passing through said window and the suspension in said compartment and to produce a signal corresponding to the optical density of the portion of said suspension therein at a fixed distance from the axis of rotation of said centrifuge, centrifuging said two portions for a time dependent on the initial solids concentration of the suspension, passing light through said windows and through said portions of the suspension, and measuring the optical densities of the two portions at said fixed distance from the axis of rotation of said centrifuge, whereby the degree of deflocculation of the original suspension can be determined.

As the rate of sedimentation of the solid particles in a suspension depends on the diameters of the particles and the difference between the specific gravities of solids and liquids, a sedimentation time is chosen so that the particles left in suspension are the ones whose degree of deflocculation it is particularly desired to measure. For example, in the case of china clay we use a centrifuging time which leaves in suspension particles of about 1 micron equivalent spherical diameter and smaller. Fine particles require more deflocculant per unit weight than coarse particles so it can be assumed that if the finest particles are deflocculated all will be. If, for example, the solids material is a crushed metallic ore having only about 20 percent by weight of particles smaller than 10 microns, the centrifuging time might be chosen so that all particles greater than about 10 microns are sedimented.

The apparatus of the invention enables there to be obtained a numerical value, which we shall designate the "deflocculation ratio," which increases as the suspension becomes more deflocculated. This deflocculation ratio is determined by the ratio of the solids concentration of the sample whose degree of deflocculation it is desired to determine to the solids concentration of the fully deflocculated sample at a centrifuging time and at a depth below the free surface chosen to give particles of the desired size in suspension at that time and at that depth. In order that the results may be self consistent, i.e. in order that a higher "deflocculation ratio" is invariably assigned to a more deflocculated suspension, slight adjustments are made to the centrifuging time dependent on the initial solids concentration of the suspension since the ratio of photocell resistances is not constant for a given ratio of solids concentrations but depends on the initial solids concentration of the suspension.

In a preferred embodiment of the apparatus of the invention, the centrifuge comprises two cooperating disc members arranged face-to-face, at least one of which is formed with recesses which constitute the separate compartments of the centrifuge. Advantageously, there are provided two truncated hollow cones arranged one within the other and coaxially with the axis of rotation of the centrifuge, one truncated hollow cone being in communication with one of said separate compartments and the other truncated hollow cone being in communication with the other of said separate compartments, the arrangement being such that said compartments can be filled with suspensions of fine particles during operation of the centrifuge. It is also advantageous if there is provided means which enable the discs to be separated whilst the centrifuge is in operation whereby the compartments can be emptied.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:

FIG. 1 is a plan view of a centrifuge for use in an apparatus according to the present invention,

FIG. 2 is a sectional elevation of the centrifuge shown in FIG. 1 taken along the line II-II of FIG. 1, and

FIG. 3 is a block diagram showing schematically one embodiment of the apparatus of the present invention.

Referring first to FIGS. 1 and 2, there is shown a centrifuge which comprises two separate sample compartments A and B. The sample compartments are defined by two discs 1 and 2, the upper disc 1 being recessed a depth of one-eighth inch to form the compartments. Except for the innermost side, the compartments are sealed by rubber sealing strips 9. The discs 1 and 2, and thus the compartments A and B, are rotated about a central axis by means of a hollow drive shaft 3. The compartments A and B can be filled, whilst being rotated, through truncated hollow cones 4 and 5 arranged one within the other. Slot 18 permits passage of material from the outer truncated hollow cone 4 to sample compartment B. Slot 19 in the wall of the outer truncated hollow cone 4 represents the exit of the tunnel from inner truncated hollow cone 5. Numeral 20 represents the end of the tunnel of square cross section which conveys material from the inner truncated cone of the sample compartment A. Each sample compartment is provided with glass windows 6 and 7 which are fitted in the upper and lower discs 1 and 2, respectively, at the same radial distances from the axis of rotation of the discs 1 and 2 i.e. with the centers of the windows five-eighths inch from the free surfaces of the suspensions, the windows in each compartment being separated by equal distances. Pairs of photoconductive cells 8 are mounted directly below the lower windows 7 in each compartment, and electrical connection to the photocells 8 is made by a slip ring assembly 14, the photocells 8 being connected into a self-balancing Wheatstone bridge network. A ring of lamps (not shown) are disposed around the truncated cones 4 and 5 and arranged so that light can be directed continuously from the lamps through the windows 6 and 7 and through the material in each compartment onto the photocells 8 during centrifuging. An overflow system comprising an overflow path P and a levelling hole 13 ensures that the compartments A and B are filled to identical levels. The upper disc 1 is connected by pillars 15 to an annular member 16 mounted below the lower disc 2. The annular member 16 supports eight symmetrically disposed hydraulic cylinders 10 each containing a piston 17 which urges the two discs together. Hydraulic fluid is introduced through the bore of the shaft 3 and passes through holes to the outer end of each of which is connected, by a compression joint, a tube leading to one of the hydraulic cylinders 10. When the oil pressure in the hydraulic cylinders 10 is released the discs separate under the action of springs 11 and the centrifuge empties into a peripheral ducting (not shown).

In operation, the discs 1 and 2 rotate at a constant speed which is generally in the range of from 1000 to 3000 r.p.m. The preferred speed is about 1500 r.p.m. which gives a sufficiently short sedimentation time; at this speed it is necessary to exert a force of 500 pounds weight on the top and bottom of compartments A and B to hold the two halves of the centrifuge together when the compartments are full. One compartment is filled with a completely deflocculated sample of the suspension and will be referred to below as the "reference compartment," and the other compartment is filled with a sample of the partially deflocculated suspension, of which it is desired to determine the degree of deflocculation, and will be referred to below as the "measurement compartment." The centrifuging time should be substantially longer than the time taken to fill the sample compartment. Thus, when the filling time is of the order of 2 or 3 seconds, the centrifuging time should be at least 2 minutes. Of the pair of photocells mounted below each window 7, one under each window is used to measure the light intensity falling on it in terms of electrical resistance; the second photocell below the window of the reference compartment is for control purposes, which will be

described below; and the second photocell below the window of the measurement compartment is a dummy included only for symmetry and balance.

As the solids concentration of a suspension in the compartments A and B increases, so does the optical obscuration of the light falling on the photocell 8. This means that if the intensity of the light directed upon the window 6 of the reference compartment is maintained constant, the electrical resistance of the photocells 8 below the window 7 will depend upon the initial solids concentration of the suspension. Although the (incident light intensity/electrical resistance) curves for the photocells 8 below the windows 7 of the two compartments are similar, they are not identical so that, with a constant illumination from the ring of lamps and a constant ratio for the solids concentrations of the suspensions in compartments A and B, the ratio of photocell resistances will depend, to some extent, on the absolute values of the solids concentrations of the suspensions in the compartments. In order to minimize this effect, the brightness of the lamp is automatically adjusted to give a substantially constant incident light intensity on the control photocell of the reference compartment, whatever the solids concentration of the suspension between the windows 6 and 7 of that compartment.

For a fixed ratio of solids concentrations of the suspension in the two compartments the ratio:

Incident light intensity on the measurement photocell decreases as the absolute values of the solids concentrations increase. This difficulty is overcome by varying the duration of centrifuging according to the initial solids concentration of the suspension being examined. Prior to commencement of centrifuging the ratio:

Solids concentration of the suspension between the windows of the reference compartment will be unity, and as the large, flocculated particles are centrifuged out of the suspension in the measurement compartment, the ratio will progressively decrease, the rate of decrease depending on the degree of deflocculation. If a suspension of high initial solids concentration is centrifuged for a shorter time, the solids concentration ratio will be higher and this will offset the reduction in the incident light intensity ratio. In order to accommodate the range of suspension solids concentration encountered in practice the centrifuging time is generally varied from about 3 to about 3½ minutes. The variable time is obtained by means of a timing unit which introduces a variable time delay into the program during centrifuging. With the lamps at full brightness, the timing unit makes a comparison of the control photocell resistance (which is dependent on the solids concentration of the suspension) with a resistance which decreases steadily with time (a potentiometer driven by an electric clock). When these resistances are in a predetermined ratio, a relay is operated to continue the program cycle. The program cycle is preferably continued at least 8 seconds and not more than 12 seconds before the end of the required centrifuging period. As centrifuging proceeds the solids concentration of the reference sample, and hence the resistance of the control photocell, falls slowly with time (as larger particles are thrown out of the suspension) and the clock-control resistance more rapidly. Also a suspension with a high initial solids concentration starts with a high photocell resistance; and the higher the initial solids concentration of the suspension, the shorter will be the time taken for the clock-controlled resistance to fall to the prescribed proportion of the control photocell resistance.

The use of the apparatus of the present invention for measuring and controlling the degree of deflocculation of a clay suspension will now be described with reference to FIGS. 1, 2 and 3 of the accompanying drawings. In FIG. 3 of the drawings, unless otherwise designated, lines bearing single arrows represent electrical connections, lines bearing double arrows represent conduits, and lines bearing triple arrows represent mechanical connections.

The apparatus shown schematically in FIG. 3 comprises a centrifuge which is constructed as described above with reference to FIGS. 1 and 2, a metering unit which regulates the supply of suspensions to the centrifuge, a program control unit which regulates the operation of the whole apparatus, and various ancillary equipment as described below.

The apparatus is connected to a conduit 31 through which there is fed a main stream of a clay suspension. Deflocculant is injected into the conduit 31 through a pipe 32. In order to measure the degree of deflocculation brought about by the injection of the deflocculant, a sample of the main stream is tapped off through a pipe 33.

A pump 34 feeds the sample to an on-off solenoid valve 35. The solenoid valve 35 is opened for a sufficient period to enable the clay suspension to flow into two metering cylinders 36 and 37 up to the level of overflow pipes. When the solenoid valve 35 is closed the clay suspension tapped off through pipe 33 is reintroduced into the main stream through pipe 33a. A pump 38 injects into the portion of the clay suspension in the cylinder 37 a quantity of deflocculant sufficient to fully deflocculate the portion, this portion thereby becoming the reference sample. After allowing sufficient time for uniform dispersion, metering overflow valves 39 and 40 are opened to allow the suspension level to fall to a lower overflow level (the reason for initially making up a larger sample than is required, is that the deflocculant dose may be metered more accurately in larger quantities).

A solenoid valve 41 is closed so that oil under pressure is delivered by a pump 58 to the hydraulic cylinders 10, thus bringing together the two discs 1 and 2 (FIG. 2) of the centrifuge to form the sealed compartments A and B (FIGS. 1 and 2).

Discharge valves 42 and 43 at the base of the metering cylinders 36 and 37 are opened to transfer the samples into their respective sample compartments in the centrifuge. When centrifuging has been in progress for about 1 minute the following operations are performed simultaneously:

- a. a ring of lamps 44 is switched on;
- b. a centrifuging time control circuit 45 is brought into operation; and
- c. an electromagnetic clutch between a servomotor and a measured value potentiometer 50 is engaged.

After a brief interval the program control unit switches off its own drive motor 46. Ten seconds before the completion of the centrifuging time, as determined by the centrifuging time control circuit 45, the drive motor 46 is switched on again. The control photocell under the window 7 of the reference compartment is switched into a brightness control circuit 47, and the brightness of the lamps is adjusted.

As a result of the different degrees of deflocculation of the samples in the compartments of the centrifuge, an out-of-balance signal from the Wheatstone bridge containing the photocells of the reference and measurement compartments is fed to a servoamplifier 48, which controls a servomotor 49 which in turn drives the measured value potentiometer 50, through an electromagnetic clutch, in a direction tending to balance the Wheatstone bridge. The following operations are then simultaneously performed:

- a. the electromagnetic clutch is disengaged so that the final position of the measure value potentiometer 50 is retained and can be recorded on a measured value recorder 59;
- b. the lamps 44 are switched off; and
- c. a timer circuit 51 and a motor 52 are switched on.

A direction control circuit 53 senses the polarity of the difference between the positions of a desired value potentiometer 54 and the measured value potentiometer 50 and selects the direction of the motor 52 accordingly whereby a flow control valve 55 is operated. The timer circuit 51 determines the magnitude of the difference between the two potentiometer positions and the motor 52 is energized for a corresponding length of time. The solenoid valve 41 is opened thus releasing the oil pressure in the hydraulic cylinders 10 and allowing the two halves of the centrifuge to separate, under the action of

springs 11, and eject their contents. A solenoid valve 56 is opened thereby allowing rinsing water to flow into the metering cylinders 36 and 37 and thence into the centrifuge. The solenoid valve 41 is opened and closed several times to allow the centrifuge to fill with water to overflowing before discharging, whereafter the solenoid valve 56 is closed. The solenoid valve 41 is then opened and the centrifuge is allowed to spin dry, thus completing the cycle of operations.

The various parts of the apparatus are brought into operation at the appropriate time by means of a number of cam operated switches 57 which, for example, energize the solenoid valves 35, 41 and 56.

We claim:

1. An apparatus comprising a centrifuge having two separate compartments wherein the improvement comprises each of the two separate compartments being provided with a window, whereby light can be passed through material present in each of said compartments, and a photocell arranged so as to intercept the light passing through said window and the material in each of said compartments and to produce a signal corresponding to the optical density of material present in each of said compartments at a fixed distance from the axis of rotation of said centrifuge, and further being provided with means for comparing the signals obtained from the photocells, said photocells being positioned to rotate with said centrifuge.
2. An apparatus as claimed in claim 1, wherein the centrifuge comprises two cooperating disc members arranged face-to-face, at least one of which is formed with two recesses which constitute the two separate compartments of the centrifuge.
3. An apparatus as claimed in claim 2, wherein there is provided means which enable the disc members to be separated whilst the centrifuge is in operation whereby the said compartments can be emptied.
4. An apparatus as claimed in claim 2, wherein there are provided two truncated hollow cones arranged one within the other and coaxially with the axis of rotation of the centrifuge, one truncated hollow cone being in communication with one of said two separate compartments and the other truncated hollow cone being in communication with the other of said two separate compartments, the arrangement being such that said two separate compartments can be filled with suspensions of fine particles during operation of the centrifuge.
5. An apparatus as claimed in claim 1, wherein one of said two separate compartments of the centrifuge has associated therewith a photocell which measures the optical density of the sample contained in said one compartment, and wherein the other of said two separate compartments of the centrifuge has associated therewith two photocells, one of which two photocells measures the optical density of the sample contained in said other compartment and the other of which two photocells acts as a "control photocell" which controls means whereby the amount of light incident on said two photocells can be adjusted to a substantially constant value which is independent of the original solids concentration of the sample contained in said other compartment.
6. An apparatus comprising a centrifuge having two separate compartments wherein the improvement comprises:
 - a. said two separate compartments including two cooperating disc members arranged face-to-face, at least one of which if formed with recesses which constitute the separate compartments of the centrifuge;
 - b. means to enable the disc members to be separated whilst the centrifuge is in operation whereby said compartments can be emptied;
 - c. each of said two separate compartments being provided with a window, whereby light can be passed through material present in said compartments, and a photocell arranged so as to intercept the light passing through said window and the material in said compartment and to produce a signal corresponding to the optical density of material present in said compartment at a fixed distance from the axis of rotation of said centrifuge;

d. means for comparing the signals obtained from the photocells; and

e. two truncated hollow cones arranged one within the other and coaxially with the axis of rotation of the centrifuge, one truncated hollow cone being in communication with one of said separate compartments and the other truncated hollow cone being in communication with the other of said compartments, the arrangement being such that said compartments can be filled with suspensions of fine particles during operation of the centrifuge.

7. An apparatus as claimed in claim 6, wherein the means which enable the disc members to be separated whilst the centrifuge is in operation comprise spring means which is held under compression, in cavities formed in the two disc members, by the action of a plurality of hydraulic cylinders each containing a piston tending to urge the two disc members together when the two disc members are to be held together, and which spring means is allowed to return to its uncompressed state, thereby separating the two disc members, by the release of the hydraulic pressure in said hydraulic cylinders when ever the two disc members are to be separated.

8. A process for measuring the degree of deflocculation of a suspension of fine particles which process comprises separating said suspension into two portions, adding to one of said

two portions a quantity of deflocculant sufficient to completely deflocculate said one portion, introducing the portions of said suspension one into each of two separate compartments of a centrifuge, said two compartments each being provided with a window, whereby light can be passed through the portion of said suspension therein, and each having associated therewith a photocell arranged so as to interrupt the light passing through said window and the suspension in said compartments and to produce a signal corresponding to the optical density of the portion of said suspension therein at a fixed distance from the axis of rotation of said centrifuge, centrifuging said two portions for a time dependent on the initial solids concentration of the suspension, passing light through said windows and through said two portions of the suspension, and measuring the optical densities of the two portions at said fixed distance from the axis of rotation of said centrifuge, whereby the degree of deflocculation of the original suspension can be determined.

9. A process as claimed in claim 8, wherein the intensity of the light passing through said windows is adjusted to give a substantially constant incident light intensity on said photocells whatever the initial solids concentration of the portion of the suspension which is completely deflocculated.

25

30

35

40

45

50

55

60

65

70

75