Oct. 18, 1960 J. K. DONOGHUE 2,956,434

APPARATUS FOR DETERMINING PARTICLE SIZE DISTRIBUTION

Filed Jan. 17, 1955 2 Sheets-Sheet 1

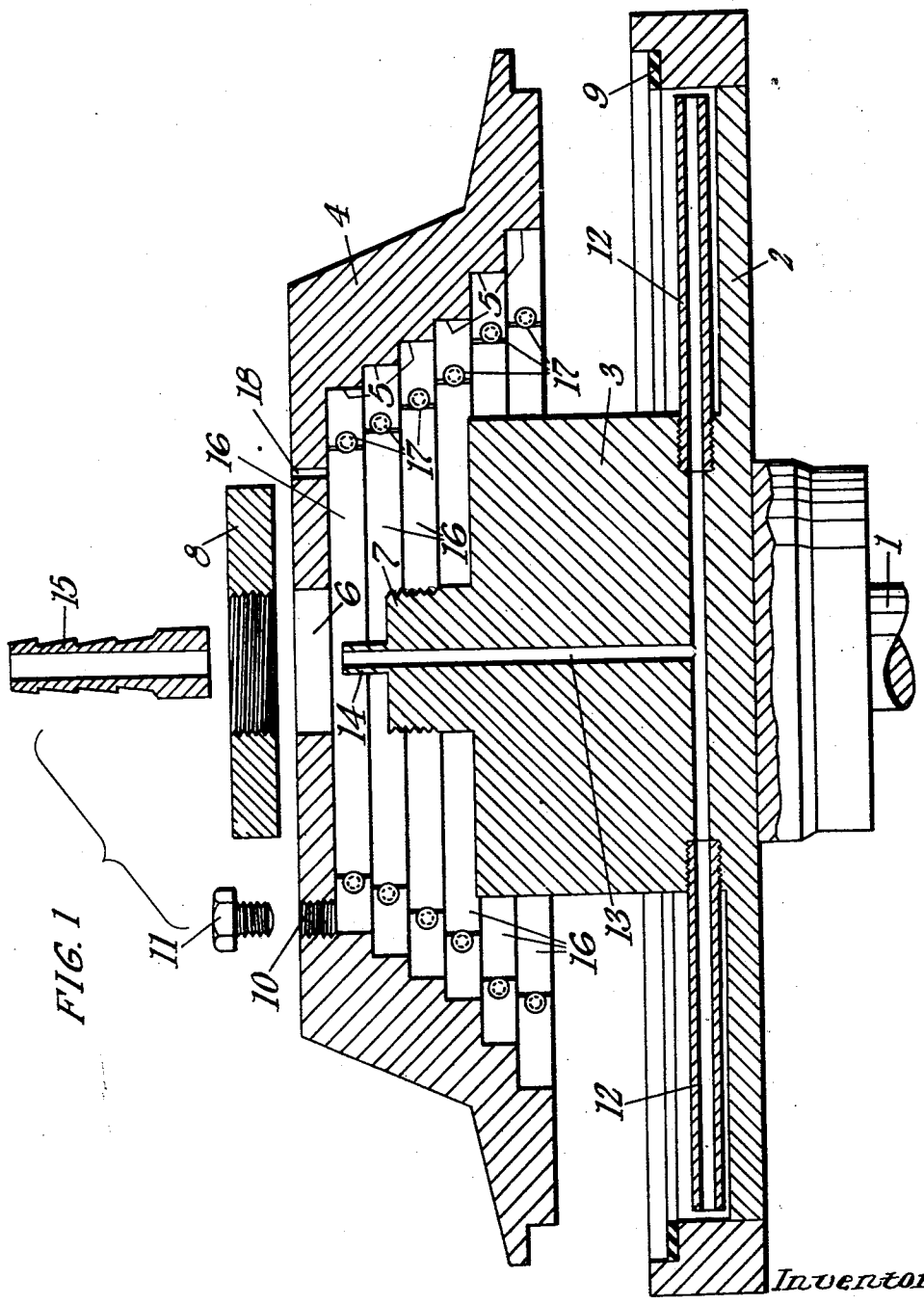



FIG. 1

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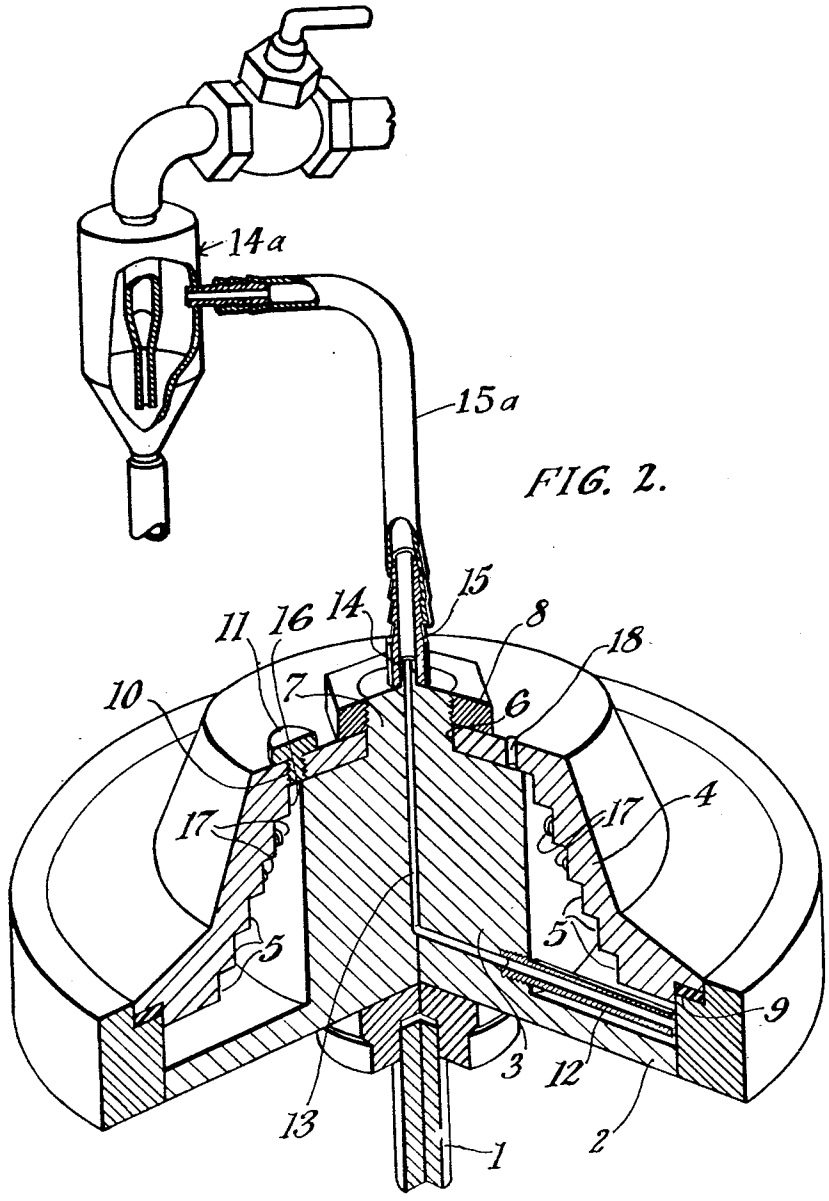



FIG. 2.

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# APPARATUS FOR DETERMINING PARTICLE SIZE DISTRIBUTION

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3 Claims. (Cl. 73—432)

This invention has for its object to provide a convenient method and apparatus for use in determining the distribution of particle size in a finely divided material, in particular a material in which all the particles are less than 5 microns in diameter.

According to the invention, a suspension of the finely divided material in a liquid of lower density than the material is centrifuged in a container having a particle receiving surface with zones of different diameters, the supernatant liquid is drawn off while the centrifuge is still running, and the quantities of material deposited on the different zones of the receiving surface are determined, from which the particle size distribution can be calculated.

The centrifuge container or rotor may be of stepped construction internally with a number of cylindrical portions of increasing diameter one below the other, the container being arranged to rotate about a vertical axis, and having a central boss concentric with the axis of rotation. Thus the liquid-containing portions have all the same internal radius but have increasing external radii, and the deposits on the upper, smaller diameter collecting surfaces will contain a larger proportion of small particles than those on the lower larger diameter surfaces.

The supernatant liquid may be drawn off by a tube extending down the central boss and having branches opening into the liquid container near the periphery of its lowest section, the tube being connected to a pump by a well-greased adaptor placed over its upper end. This arrangement is found to remove the greater part of the liquid without disturbing the deposited particles. Then the container is rotated for a short time at a higher speed to pack down the deposit, after which the remaining liquid can be drained off. Each cylindrical part of the container may have at its lower edge a lip to assist in retaining the deposit. The deposit may be collected for weighing on strips of filter paper or metal strips placed within the container.

Only a single centrifuging operation is required to obtain all the deposits ready for weighing, and the method is applicable to materials containing particles less than 5 microns in diameter, which cannot conveniently be dealt with by gravitational sedimentation owing to the slow rate of settling of the fine particles. Mathematical methods are available for evaluating the results obtained by weighing the deposits.

The invention is illustrated, by way of example, in the accompanying drawings in which:

Fig. 1 is an axial section through a centrifuging apparatus embodying a preferred form of the invention, and

Fig. 2 is a perspective of the centrifuging apparatus of Fig. 1, parts being broken away to show the interior construction.

Referring to the accompanying drawing, which shows the separate parts of a centrifuging apparatus in accordance with the invention in section, a motor shaft **1** has mounted on it a lower rotor part **2** having a central cylindrical boss **3**. The upper rotor part **4** has a stepped



inner surface with a number of cylindrical portions **5** of increasing diameter one below the other. It has a central hole **6** to fit over a projection **7** on the boss **3** and is secured in position by a ring **8** screwed on to the projection **7**. Its outer edge is sealed against that of the lower part **2** by a packing ring **9**. There is an inlet hole **10** in the top of the upper part **4**, which is closed by a plug **11**.

The boss **3** has radial tubes **12** screwed into it beneath the part **4** with their open ends close to the inner surface of the part **2**, and ducts **13** in the boss **3** lead from the tubes **12** to an outlet tube **14** at the top of the projection **7**, to which a water pump **14***a* may be attached by an adaptor **15** and tube **15***a*. On each of the stepped surfaces **5**, two metal strips **16** are held between pins **17**. Each strip has a length equal to one-quarter of the circumference of the corresponding stepped surface **5**, and the strips on each stepped surface are diametrically opposite each other, on a diameter at right angles to that passing through the tubes **12**. There is a small air inlet hole **18** in the top of the part **4**.

The rotor is filled with a liquid containing in suspension a sample of the material to be examined, through the inlet hole **10**, and the hole closed by the plug **11**. The rotor is then rotated at centrifuging speed for the desired time, after which the liquid is withdrawn through the tube **14** while the rotor is still rotating, air entering through the hole **18**. Then the rotor is rotated at a high speed for a short time to pack down the deposit, and after it has been stopped the metal strips **16** are removed, dried and weighed. In the case of a rotor having a capacity of 1350 ml. and running at 480 r.p.m. the liquid can be withdrawn in about 30 seconds. The running time for the test may be between 15 and 120 minutes.

The fractional weights $p$, deposited, i.e. the weights of the deposits expressed as fractions of the weights of material initially contained in the respective layers, are plotted against the radii, R, of the receiving surfaces, and from the $p—R$ curve so obtained a $dp/dR—R$ curve is derived. By suitable mathematical treatment of the data obtainable from these curves, it is possible to calculate the percentage weight of the particles in the original sample which are of more than a given diameter.

If desired the centrifuge may be arranged with the rotor axis horizontal instead of vertical. The centrifuge with vertical rotor axis may be arranged with the smallest stepped surface at the bottom and the largest, together with the withdrawal tubes, at the top. Any convenient number of stepped surfaces may be used.

It is further possible to use instead of a rotor of stepped construction, one having a receiving surface with a continuous slope, this being equivalent to a series of infinitely small steps. The particles are deposited on a sheet placed on the sloping surface, which is removed for evaluating the deposits. For this purpose the sheet may be cut into strips which can be weighed, or the weight distribution may be determined by an optical method, the transmission of light through the sheet carrying the deposit at any point being used as a measure of the weight deposited at that point. Mathematical analysis of the way in which the particles in the suspension move in the centrifugal field shows that the particle size distribution can be calculated from the relationship between the fractional weight $p$ deposited on a collecting strip and the radius R of the collecting strip. The formula giving the fraction of the material having a diameter greater than a value D is:

$$\text{Percent oversize} = p + \frac{R(R^2 - S^2)}{2S^2} \frac{dp}{dR} \qquad \text{(Equation 1)}$$

where S is the radius of the centre boss **3** D is determined from the time ($t$) of running the centrifuge, a constant

$k$ (which is related to the difference in density of the liquid ($d$) and solid ($e$), the angular velocity of the centrifuge ($w$) and the viscosity of the liquid ($n$)):

$$k=\frac{(e-d)w^2}{18n} \qquad \text{(Equation 2)}$$

and the ratio of R to S:

$$D^2=\frac{1}{kt}ln(R/S) \qquad \text{(Equation 3)}$$

In practice, the weight of material deposited on the collecting strips enables the curve of $p$ against R to be drawn. A value of D is then assumed and from the formula for D (Equation 3), using the known parameters of the particular test, the corresponding value of R is calculated. The slope

$$\frac{dp}{dR}$$

of the $p-R$ curve is then measured at that value of R and the appropriate values substituted in Equation 1. The procedure is repeated for the assumed values of D, until a full particle size distribution is built up.

What I claim is:

1. The method of determining the distribution of particle size in a finely divided material comprising the steps of introducing a suspension of the finely divided material in a liquid of lower density than the material into a container having a particle receiving surface with zones of different diameters, centrifuging the suspended material at a predetermined distributing speed, withdrawing the substantial part of the supernatant liquor while centrifuging at the said distributing speed, centrifuging the remaining suspension at a higher pack-down speed, determining the quantities of material deposited on the different zones of the receiving surface and calculating the particle size distribution from these determinations.



2. Means for determining the distribution of particle size in a finely divided material which is suspended in a liquid of lower density than the finely divided material, comprising a centrifuge with a rotatable container having a particle receiving surface, a central cylindrical boss surrounded by the particle receiving surface, the particle receiving surface having zones of different diameters, liquid withdrawal tubes opening near the circumference of the interior of the container where its diameter is greatest, and an outlet tube with which the withdrawal tubes communicate, adapted to be rotatably connected to suction means for withdrawing centrifuged liquid.

3. Means as claimed in claim 2, and having strips of sheet material removably attached to the zones of the container, for receiving the deposited particles.

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