No. 809,959.

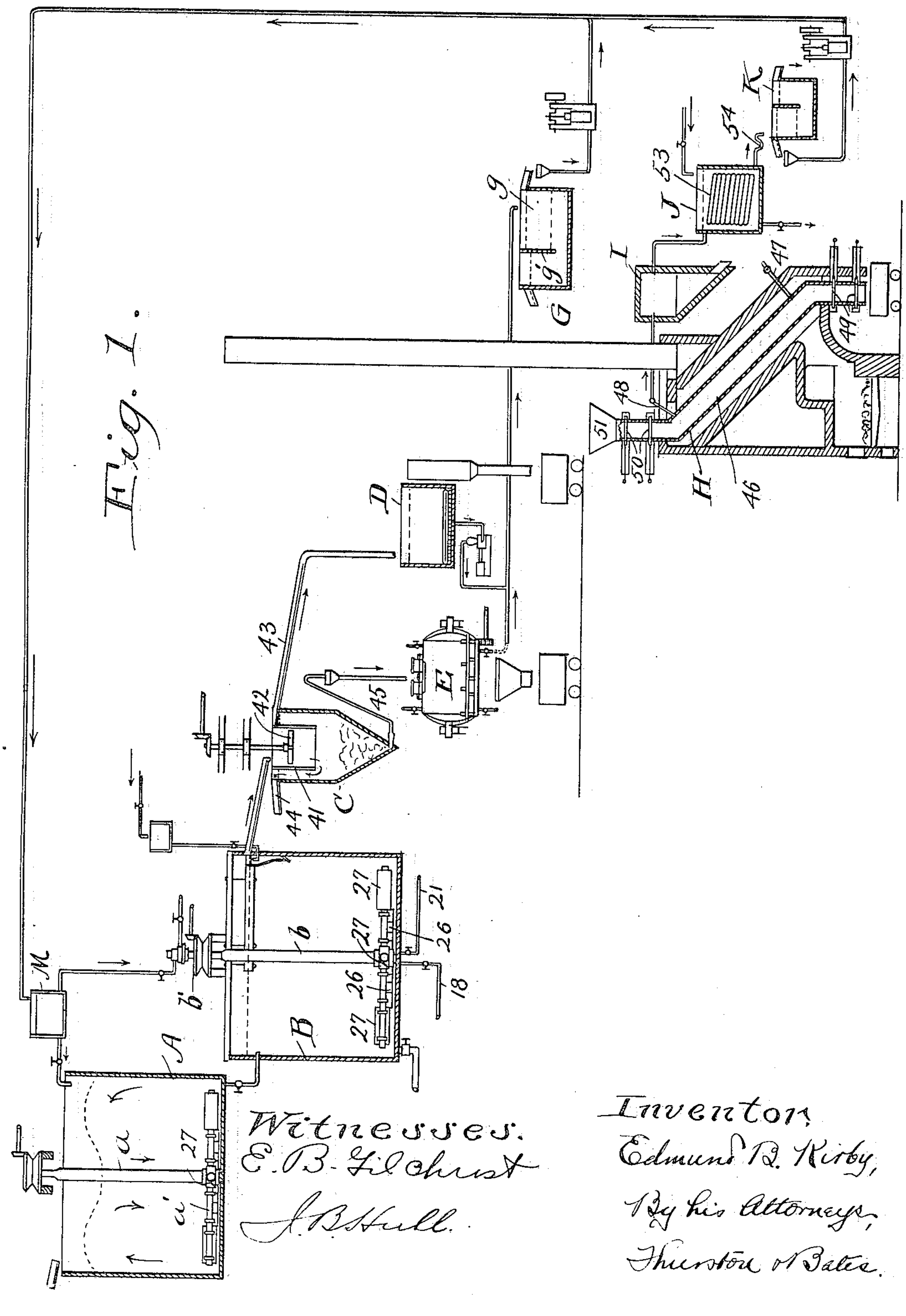
PATENTED JAN. 16, 1906.

E. B. KIRBY.

PROCESS OF SEPARATING MINERALS.

APPLICATION FILED DEC. 14, 1903.

3 SHEETS—SHEET 1.



Witnesses:
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Inventor,
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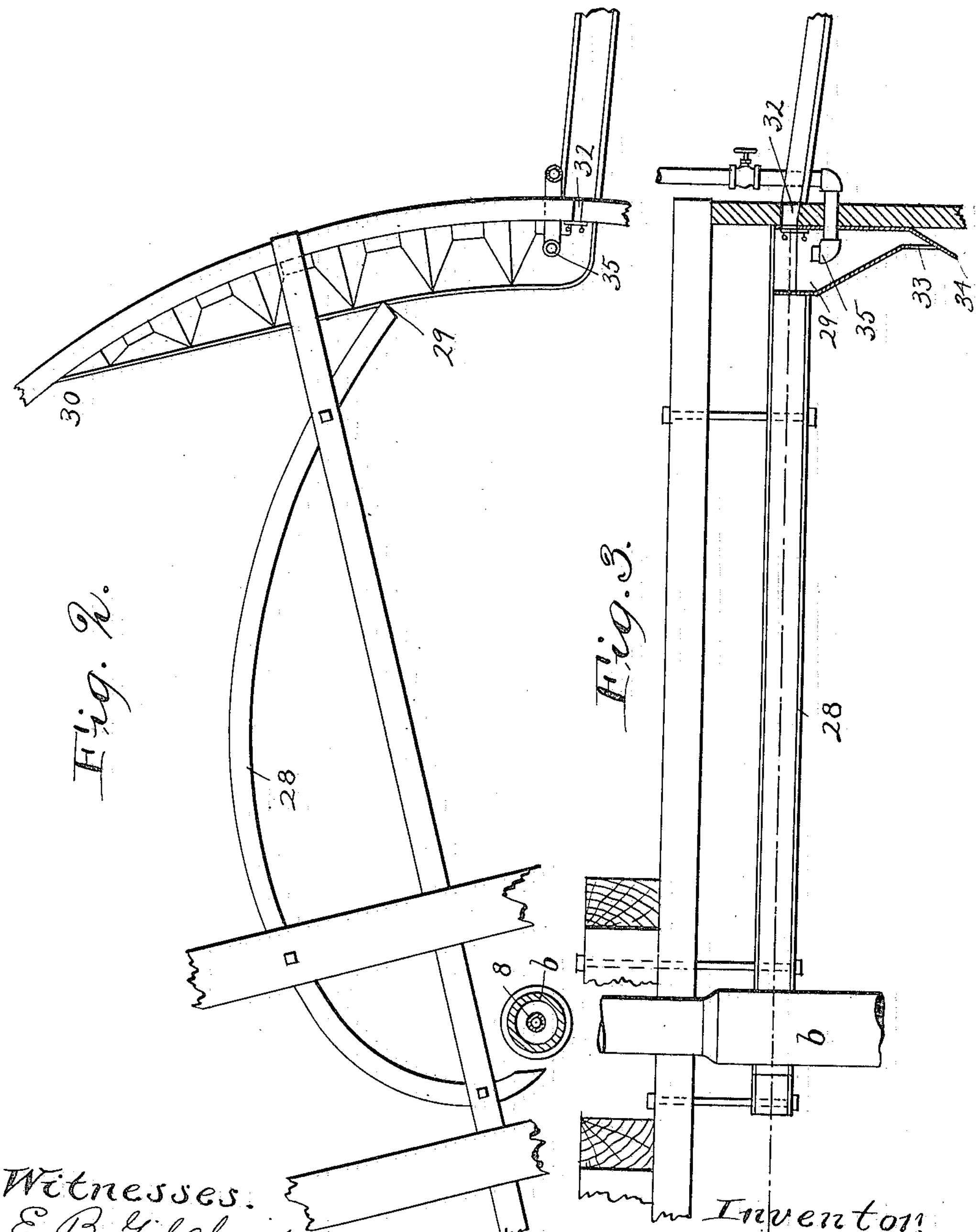

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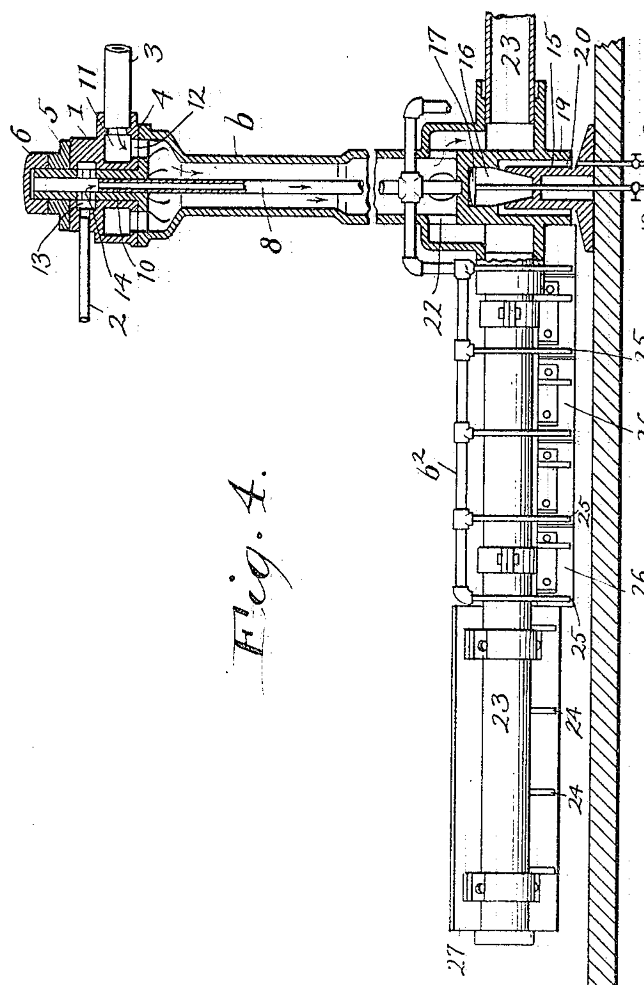

Fig. 4.

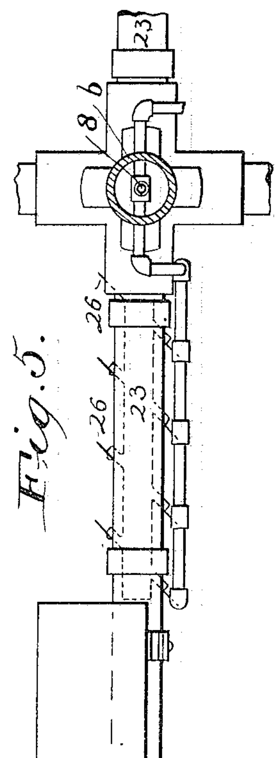

Fig. 5.

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UNITED STATES PATENT OFFICE.

EDMUND B. KIRBY, OF ROSSLAND, CANADA.

PROCESS OF SEPARATING MINERALS.

No. 809,959. Specification of Letters Patent. Patented Jan. 16, 1906.

Application filed December 14, 1903. Serial No. 186,033.

To all whom it may concern:

Be it known that I, EDMUND B. KIRBY, a citizen of the United States, residing at Rossland, in the Province of British Columbia and Dominion of Canada, have invented a certain new and useful Improvement in Process of Separating Minerals, of which the following is a full, clear, and exact description, reference being had to the accompanying drawings.

The invention relates to the concentration of ores. It may be employed to separate the metallic minerals from the gangue, or to separate certain of the metallic minerals from others, or from others and the gangue.

The operation of the process is dependent upon the fact that, because of differences in physical characteristics of the various constituents of mineral material, such constituents show preferences of adhesion between two commingled, but immiscible liquids.

The invention consists in the process hereinafter described, and in the several steps thereof, all of which will be definitely set forth in the claims.

In the drawings, Figure 1 is a diagrammatic view of an apparatus with which the process may be practiced, the several parts thereof being shown in vertical section. Fig. 2 is a plan view of one side of the separating-tank. Fig. 3 is a vertical sectional view of the same mechanism. Fig. 4 is an elevation of a part of the agitator mechanism of the separating-tank, some of said mechanism being in section; and Fig. 5 is a plan view of one of the agitator-arms and a sectional plan view of the shaft.

The process is applicable to a great variety of ores, and may be practiced in many cases so as to separate those metallic minerals which must be treated in one way from those which must be treated in some other way in order to recover their contained commercial metals, and since it is capable of being used for this purpose as well as to separate the metallic minerals from the gangue it is thought to be a great step in advance of this art.

It is believed that the process or some of the novel steps thereof may be employed with advantage in the treatment of all ores. It is obviously impossible, however, to give definite directions for attaining the best results with all ores, because of the great chemical and physical differences which they exhibit. The detailed directions hereinafter set forth are those which are found most efficient in the treatment of the Rossland, British Columbia, ores, with which I have done the most work, for the primary purpose of separating the chalcopyrite (which must be smelted) from the other constituents which may be subjected to other after treatment for the recovery of their contained metals.

The process, as an entirety, in its best form for use with Rossland ores for the purpose stated consists in the following steps:

First, in thoroughly agitating together (*a*) the pulverized ore or mineral material (*b*) enough water to make with said pulverized ore a flowing pulp, and (*c*) a solution of bitumen in a thin distillable hydrocarbon liquid as kerosene, these materials to be so thoroughly agitated together as to finely subdivide said solution into small globules and bring said globules into contact with substantially all of the pulverized mineral particles which will, by preference, adhere to them.

Second, in allowing the hydrocarbon-coated particles to float to the surface of the mass, and in rendering this separation substantially complete by gently agitating the mass, and by injecting gas into the same, and preferably also discharging into the mass fine streams of the solution. When the separation is completed, the floating hydrocarbon-coated concentrate is removed for subsequent treatment.

Third, in filtering said concentrate to free it so far as possible from the hydrocarbon liquid.

Fourth, in distilling said coated concentrate and condensing the hydrocarbon vapor to be used again.

It is thought that the use of a gas to assist in the flotation of the coated particles, as set forth in the description of the second step of the process, is radically new in this art, irrespective of its association with the other steps described. It is that which makes it possible for the first time to use thin oils and hydrocarbon. The prior processes which use thick, viscous oils will, however, be much aided by the addition of this step, because in spite of all the care which is exercised in the practice of those processes to keep the oil in large clots or masses a great deal of it is "floured" or broken up into minute particles which are trapped in the sands and lost. The employment of the gas in the manner stated brings in a more powerful floating agency than anything before used, which results in the recovery of this floured oil together with numerous coated particles which would not otherwise be floated. This step of the process is therefore useful with any and all liquids lighter than water which exhibit preference of adhesion for the metallic mineral particles. Kerosene alone, for example, may be used with most ores to take out the sulfids provided the gas is used, as stated, to cause the flotation of the kerosene-coated particles. It is the bitumen, however, dissolved in the kerosene which gives the precise adhesive preference which enables it to separate the gold and chalcopyrite from the crushed ore. The bitumen may be asphalt or the bitumen produced by the distillation of petroleum to a semisolid residuum, or it may be tar, pitch, or any other specific form of bitumen. In treating Rossland ores I have found that the most satisfactory results have been attained by using a solution obtained by dissolving in kerosene about five per cent. or thereabout of Trinidad asphalt or the semisolid residuum of petroleum distillation. Preferably the pulverized ore is mixed with three to five times as much water, by weight, and to this is added a sufficient amount of the kerosene-bitumen solution, excellent results being obtained by using one-fourth to three-fourths as much, by weight, as ore. The preference of the solution for some of the mineral particles may be regulated by altering or varying the quantity of the solute substance and by varying the temperature at which the solution is used. The preference of the water for other mineral particles may be regulated by adding some acid or other chemical. A distinct advantage of using a light hydrocarbon, like kerosene, is that so much of it as cannot be removed from the concentrate by mechanical means may be recovered by a process of distillation, this method of recovery being impossible when thick non-distillable oils are used. The injection of a gas, preferably air, into the mass, which is the chief novel characteristic of the second step of the process, assists in the flotation of the hydrocarbon-coated particles. This makes it possible to finely subdivide the solution by the agitation, and this greatly increases the chance that all of the mineral particles which exhibit preferential adhesion for it shall be brought into contact with it. Some of the hydrocarbon-coated particles will float to the surface without assistance; but a considerable quantity of such particles will not be sufficiently buoyant, and some of such particles and some globules of the mixture would be trapped in the sands. In order to recover this less buoyant material together with the globules of the mixture, the mass which tends to settle is slowly lifted and turned over to liberate the coated particles and the globules, and at the same time a gas, preferably air, is blown into the mass preferably near the bottom thereof. The air-bubbles not only tend to attach themselves directly to the coated particles, and thus float them to the surface, but the air becomes dissolved in the water to its maximum capacity. This dissolved air tends to again separate itself from the water and attach itself in minute globules to the coated particles. I find that air, carbon dioxid, hydrogen, and marsh-gas are satisfactory for this purpose, and doubtless many other or all gases will operate in the same way, but I prefer air. It might be here added that because the solution is broken up into small globules there is little likelihood that any of the non-coated particles shall be entangled with the coated concentrates and carried to the surface. In removing this floating hydrocarbon-coated concentrate it is practically impossible to exclude some of the water in which the non-coated particles are held in suspension. It is therefore desirable that this concentrate shall be washed free from such non-coated minerals. This step is not, however, absolutely essential. It is possible to remove a very large part of the hydrocarbon by filtration, because of its thin character. It is not, however, possible to remove it all by this or any other mechanical process; but because of the character of the hydrocarbon solvent used it is possible to recover all of it for future use by a process of distillation which constitutes the fourth step of the complete process.

I will now describe the apparatus shown in the drawings for practically carrying on said process.

A represents the mixing-tank.

B represents the separating-tank.

C represents the settling-tank.

D represents the filter, and E another filter, which may or may not be used according to circumstances.

G represents a settling box or tank into which the liquid from the filter or filters is discharged.

H represents the retort-furnace; I, the dust-collector used in connection therewith; J, a condenser; K, the settling-tank, in which the condenser discharges the condensed vapor, and M represents a reservoir from which the solution may be fed into the mixing-tank and into the separating-tank.

In the separating-tank B is a vertical shaft *b*, having on its upper end a gear *b'*, by which it may be rotated. The head of the shaft above the driving-gear passes through an oil and air box 1, which remain stationary while the shaft revolves and is supplied by the hydrocarbon-pipe 2 and the air-pipe 3. The box rests on the rotating shoulder 4 of the shaft and is held down to a tight joint by the collar 5 and lock-nut cap 6. Through the hollow shaft 7 extends a small hydrocarbon-pipe 8, the upper end of which is firmly inserted within the upward extension 10 of the shaft, and this central pipe conveys the hydrocarbon. In the stationary box the air enters the annular chamber 11, passing through its open bottom through a set of apertures 12 to the interior of the shaft. The hydrocarbon enters a similar annular chamber 13, from which it passes by apertures 14 into the top of the central hydrocarbon-pipe. The hub and revolving arms are shown in Figs. 4 and 5. The step-bearing is supported on a pedestal 15, provided with a wooden block 16, which supports the moving wearing-plate 17 of the shaft. Lubricating-water under pressure is introduced through the pipe 18, finding its way out from the bearing through side grooves 50. The sides 19 of the shaft are carried down below the bearing, so as to leave an annular space 20 between them and the pedestal. This annular space is intended to constitute an air-bell designed to assist the lubricating-water in excluding sand from the bearing. The air-supply in it is maintained by a slight stress of air which escapes beneath the bell through the pipe 21. The air-current for the charge passes down through the shaft, passing (as shown by the arrows) through the side channels 22 into the hollow arms 23 23. From each arm it passes out through drop-pipes 24 24. The hydrocarbon liquid is delivered through small pipes parallel with said hollow arms, emerging at the outlets 25 25. The radially-scraping blades 26 26 are secured to the arms 23 and also the inclined lifting-plow 27 at the extremity of the said arms, this being so set as to force the circulation upward at this point. The rotary movement of the charge leads the floating scum of hydrocarbon liquid, air-bubbles, and concentrates against the curved skimming-bar 28, which is hung so as to arrest and deflect this floating layer and cause it to pass into the settling and washing chamber or box 29. The edge of this box outside of the skimming-bar is submerged sufficiently to allow the floating material to pass over it, while the remaining part of said edge is raised above the liquid, so as to detain everything passing into it. Owing to the agitation within the tank caused by the movement of the arms 23 and the rising air-bubbles the water, even near the top, is not clear, but turbid or muddy, with slimes or fine particles of the non-coated minerals which do not settle rapidly enough to get out of the way. The floating concentrates are carried mainly at the lower surface of the hydrocarbon layer, where it is in contact with the water. The discharge-gate 32 in order to permit these floating particles to pass out must be set low enough to clear them, and must therefore allow a portion of the water to pass out with the skimmings, and this muddy water would therefore carry its suspended particles of the worthless minerals, which would make the concentrates impure. The settling and washing chamber or box is designed to lessen or prevent this evil. As the floating material passes over its submerged edge 29 30 it escapes from the swift current and rising air-bubbles, so that in its comparative quiet the slimes have a better opportunity to settle out of the way. The bottom of the box is divided as convenient into compartments by submerged partitions, as shown, each compartment terminating in a hopper-shape bottom with discharge-openings 33, through which the settled slimes may pass out again into the tank. Projecting shields 34 prevent the air-bubbles from entering the hoppers and disturbing their quiet. The passage of the floating material over this quiet chamber or box settles most of the slime before reaching the adjustable discharge gate or outlet 32. Before reaching the gate, however, the skimmings pass over a stream of clean wash-water introduced at the point 35 through the pipe shown. This wash-water is delivered under constant head from a supply-tank. Its quantity is made exactly equal to that passing out through the discharge-gate with the skimmings, so that this discharge, being supplied entirely by the pure water close at hand, contains little or none of the muddy water which is thus held back in the tank. It is evident that the incoming and outgoing streams are self-adjusting, because if too much enters the general level rises and a larger stream flows from the orifice.

In the mixing-tank a vertical rotating shaft a is mounted having, preferably, a lower bearing similar to that which is provided for shaft b and which has been described. Arms a' are attached to the shaft near its lower end, and lifting-plows are secured to the outer parts of these arms and radial plows to the other parts thereof, just as in the separator-tank. This shaft a is to be rotated rapidly, and the result is a thorough commingling of the various parts of the charge, which result is facilitated by the currents created in the charge by the action of said plows, the direction of said currents being indicated by the arrows in Fig. 1. This separate-tank for performing the mixing operation is not necessary for my process, although it is preferable in some cases, as when a continuous discharge is desired. The mixing may be performed just as well in the separating-tank, which may then be termed the "mixing" and "separating" tank. It is merely necessary to rotate the agitating mechanism rapidly while mixing and to rotate it slowly while the separation is being made.

The material skimmed from the surface of the separating-tank may pass directly to the filter D or E; but it is best to discharge it into the settling-tank C in order to separate the main bulk of water, and thus reduce the bulk of liquid to be put through the filtering operation. As explained, this is not essential, but is conveniently introduced prior to the filtration merely in order to separate the main bulk of water, and thus reduce the bulk of fluid to be put through the filtering operation. It is thus merely a convenience. The settling-tank C is shown as a cylindrical vessel with hopper-shape bottom, within which is suspended a cylinder 41, reaching half-way down. The stream of skimming enters this central cylinder, within which the water and hydrocarbon separate, the former sinking, while the latter, with its accompanying concentrates and air-bubbles, floats in a layer, as shown. The stirring apparatus 42 has its arms revolving gently within this layer, so as to break up and discharge air-bubbles and assist the separation. Most of the concentrates hang near the contact between the hydrocarbon and water, and as this contact-surface becomes overloaded with concentrates some of them sink to the bottom of the tank. The excess of hydrocarbon, accompanied by some of the concentrates, flows out through the launder 43, while the excess of water passing beneath the suspended internal cylinder passes out through the overflow 44, and thereby lessens the volume to be filtered. The concentrates which fall to the bottom of the tank, accompanied by the hydrocarbon which adheres to them, are drawn off in a thick condition through the pipe 45, the discharge end of which is raised to prevent the exit of more water than necessary. The two streams, one of hydrocarbon and concentrates, the other mainly of water and concentrates, may either be filtered in separate apparatus or united and put through the same apparatus, as is found most convenient. Hydrocarbon liquid and concentrates filter more easily than when water is present, and it may therefore be desirable to filter separately. It is for this reason that two filters D and E are shown, the one receiving such material as flows from the surface of the tank through launder 43 and the other that material which is discharged through pipe 45. I do not restrict myself to any particular form of filtering appliance.

The use of thin hydrocarbon liquid in place of the thick viscid oils used by other inventors makes filtration comparatively easy and permits the use of more simple and cheap methods than the centrifugal machine or filter-presses. The use of such simple apparatus is also made possible by the fact that it is not now necessary to separate the liquid very thoroughly, since its extraction is to be perfected in the distillation-retorts. I find that in some cases it is sufficient to use a simple open filter-tank with a porous bottom of any of the well-known kinds, preferably light canvas, resting on suitable supports. The liquid and water drain through the porous bottom, leaving the concentrates in the tank sufficiently drained to be shoveled into the retorts. The passage of the liquid and water through the porous bottom is aided by the well-known means of a vacuum-pump N beneath. The filter (indicated by E) is a pressure-filter barrel of well-known construction, which need not be here explained, and may be used when forced filtration is necessary. It is shown to emphasize the fact that the operation of the process is not restricted to any specific kind of filter or to any number of filters. The mixture of hydrocarbon liquid and water now free from solid matter is of course self-separating in any receptacle. A convenient form is shown in the settling-box G, into which said mixture is delivered through suitable pipes from both filters. The hydrocarbon liquid remains in one compartment g, from which it overflows and is returned to the reservoir. The water sinking to the bottom passes under the partition g' to the other compartment and flows to waste. The filtered concentrates containing some residual liquid and moisture are now ready for the distilling operation for the recovery of the five per cent. or more of valuable hydrocarbon liquid remaining in them. This is done in the retort-furnace H. An iron retort 46 is set in a furnace at such an angle that the concentrates will pass down through it by gravity, but will not altogether close the upper side of the channel, which should remain more or less open for the exit and passage of the steam and hydrocarbon vapors. The retort is maintained at the distillation temperature of the hydrocarbon used, which in the case of kerosene is about 338° Fahrenheit. In order to assist in carrying off the hydrocarbon vapor, a current of superheated steam is introduced at 47, while the steam and hydrocarbon vapors pass off through the pipe at 48 to the condensing apparatus. The dry concentrates on losing their liquid and moisture slide down to the lower end of the retort, where they are drawn off through the double gates 49 49, which are opened alternately, so as to prevent the escape of vapors. As concentrates are drawn off below a fresh mass is introduced above by the alternate opening of the gates 50 50, which likewise prevent the escape of vapors. An additional seal is provided by the hopper 51, which is kept filled with concentrates. The steam and hydrocarbon vapors may be led through a dust-collecting chamber I, designed to settle and collect any concentrates dust carried over, and then passes to the condenser J. This includes a metal worm or coil 53, set in a tank through which a stream of cooling-water is allowed to flow. The condensed hydrocarbon and water passing through a U-trap 54 flow into a settling-box K, similar to the one described at G. Here the hydrocarbon liquid and water separate, and the former is returned to the reservoir M for reuse, as is also the liquid recovered in the settling-tank G.

Having described my invention, I claim—

1. The process of separating minerals, which consists in mixing together pulverized mineral material, a considerable quantity of water, and a substance immiscible in water, but of less specific gravity, and which will, in the presence of water, adhere to some of the mineral particles and not to others; in violently agitating the mass so as to break up said immiscible substance into minute globules; in allowing said mass to settle whereby a considerable quantity of the mineral particles having become coated with said substance will float to the top of the mass, and in gently agitating the portion thereof which settles, and in blowing into the same a gas for the purpose of assisting the flotation of said substance and the mineral particles coated therewith; in removing the floating layer, and in separating the mineral particles from said immiscible substance, substantially as specified.

2. The process of separating ores, which consists in mixing together pulverized mineral material, a considerable quantity of water, and a solution of bitumen in a light hydrocarbon liquid, the proportion of bitumen in solution being substantially sufficient to insure the coating and entrainment of the mineral particles; in violently agitating this mixture to break up said solution into fine globules; in allowing the mass to settle, and then gently agitating the same and blowing in gas to insure the flotation of said solution and the mineral particles coated thereby; in removing the floating layer; and separating the mineral particles from the solution, substantially as specified.

3. The process of separating ores, which consists in mixing together pulverized mineral material, a considerable quantity of water, and a solution of bitumen in a light hydrocarbon liquid, the proportion of bitumen in solution being substantially sufficient to insure the coating and entrainment of the mineral particles; in violently agitating this mixture to break up said solution into fine globules; in allowing the mass to settle, and then gently agitating the same and blowing into it a gas and some of the said solution to insure the flotation of said solution and the mineral particles coated thereby; in removing the floating layer; and separating the mineral particles from the solution, substantially as specified.

4. The process of separating minerals, which consists in mixing together the pulverized mineral material a considerable quantity of water, and a solution of bitumen in a light hydrocarbon liquid; in allowing the same to settle, and removing therefrom the floating layer of said solution and the mineral particles which have been coated thereby; and in filtering the material so removed, substantially as specified.

5. The process of separating minerals, which consists in mixing together the pulverized mineral material, a considerable quantity of water, and a solution of bitumen in a light hydrocarbon liquid, the proportion of bitumen in solution being substantially sufficient to insure the coating and entrainment of the mineral particles; in gently agitating this mixture and blowing gas into the same to assist the flotation of said solution and the mineral particles which have been coated thereby; in removing said floating layer; and filtering the same, substantially as specified.

6. The process of separating minerals which consists in mixing together the pulverized mineral material, a considerable quantity of water, and a solution of bitumen in a distillable hydrocarbon liquid, the proportion of bitumen in solution being substantially sufficient to insure the coating and entrainment of the mineral particles; in allowing the same to settle, and removing therefrom the floating layer of said solution and the mineral particles which have been coated thereby; in filtering the material so removed; and in distilling the concentrate residue and condensing the hydrocarbon vapors driven off, substantially as specified.

7. The process of separating minerals, which consists in mixing together the pulverized mineral material, a considerable quantity of water, and a solution of bitumen in a distillable hydrocarbon liquid; in gently agitating this mixture and blowing gas into the same to assist in the flotation of said solution and the mineral particles which have been coated thereby; in removing the floating layer, and filtering the same; and in distilling the concentrate residue and condensing the hydrocarbon vapors driven off, substantially as specified.

8. The process of separating minerals, which consists in mixing together the pulverized mineral material, a considerable quantity of water, and a solution of bitumen and kerosene; in gently agitating this mixture, and in blowing a gas into the same to assist in the flotation of said solution and the mineral particles which have been coated thereby; and in separating said solution and mineral particles, substantially as specified.

9. The process of separating minerals, which consists in mixing together the pulverized mineral material, a considerable quantity of water, and a solution of bitumen in kerosene, and in vigorously agitating this

mixture so as to break up said solution into minute globules; in gently agitating said mixture and blowing a gas into the same to assist in the flotation of said solution and the mineral particles coated thereby; in removing the floating layer; in washing and filtering the same; and finally in distilling the concentrate residue and condensing the hydrocarbon vapors driven off, substantially as specified.

In testimony whereof I hereunto affix my signature in the presence of two witnesses.

EDMUND B. KIRBY.

Witnesses:

T. L. SAVAGE,

E. G. EASTMAN.