

Oct. 15, 1940.

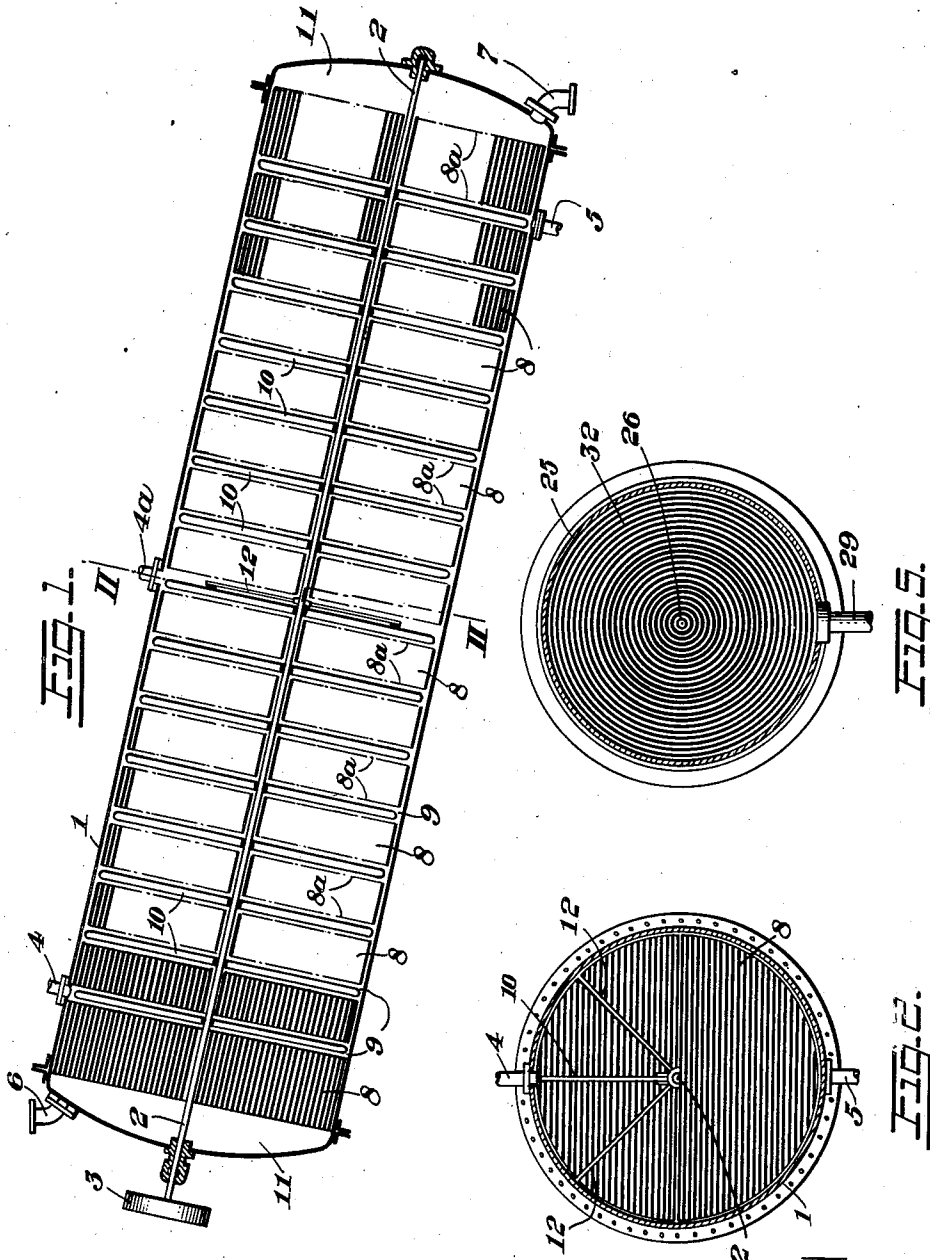
G. BOTTARO

2,218,080

PROCESS FOR COUNTERCURRENT EXTRACTION

Filed May 23, 1936

4 Sheets-Sheet 1



Inventor.
GIACOMO BOTTARO
By *Haseltine, Lake & Co.*
Attorneys

Oct. 15, 1940.

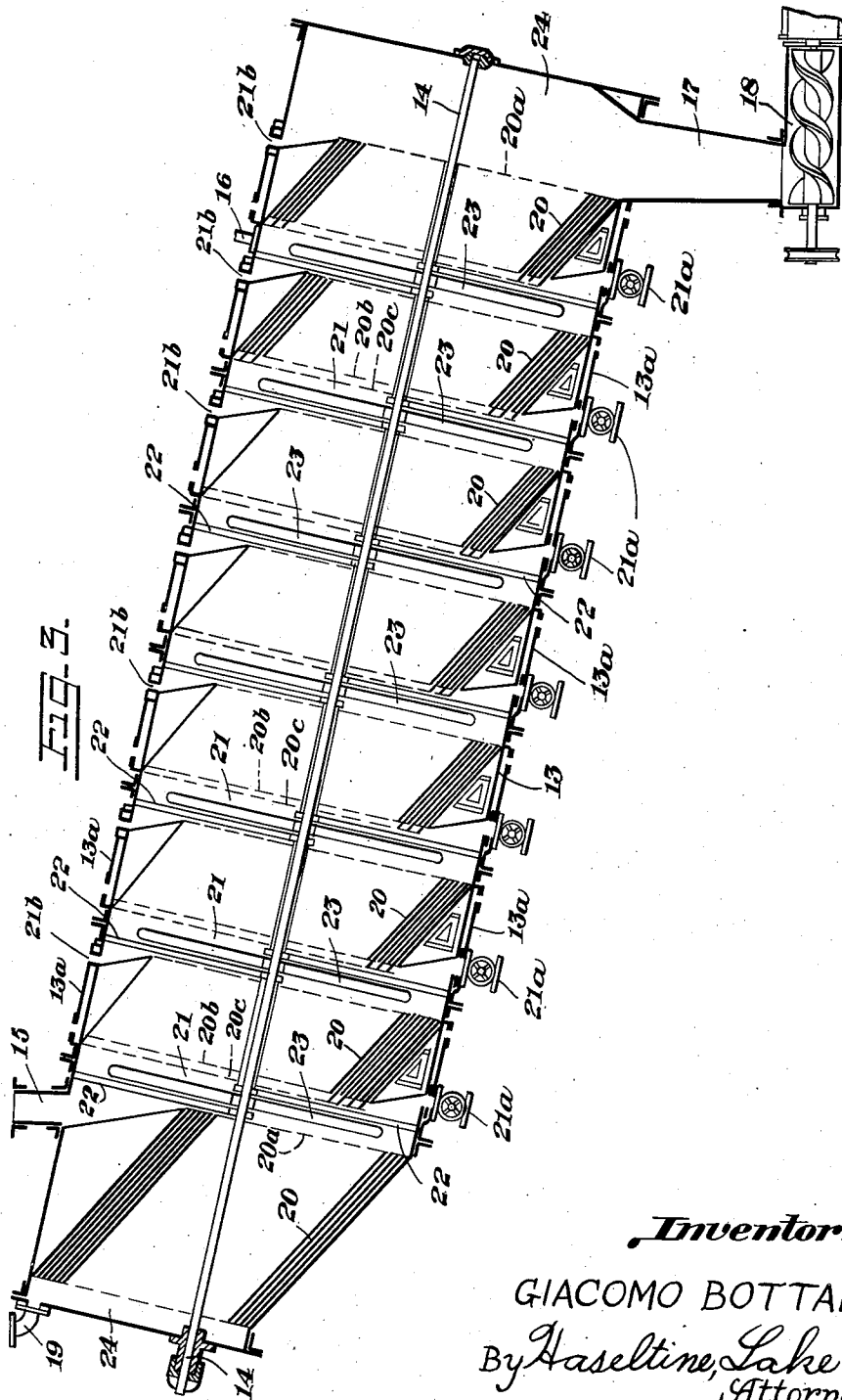
G. BOTTARO

2,218,080

PROCESS FOR COUNTERCURRENT EXTRACTION

Filed May 23, 1936

4-Sheets-Sheet 2



Inventor.

GIACOMO BOTTARO
By *Haseltine, Lake & Co.*
Attorneys

Oct. 15, 1940.

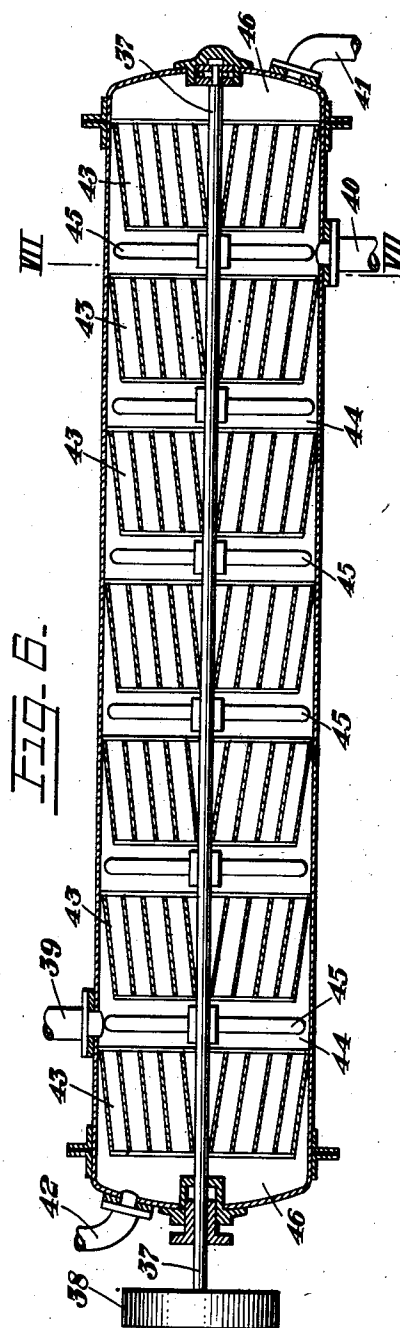
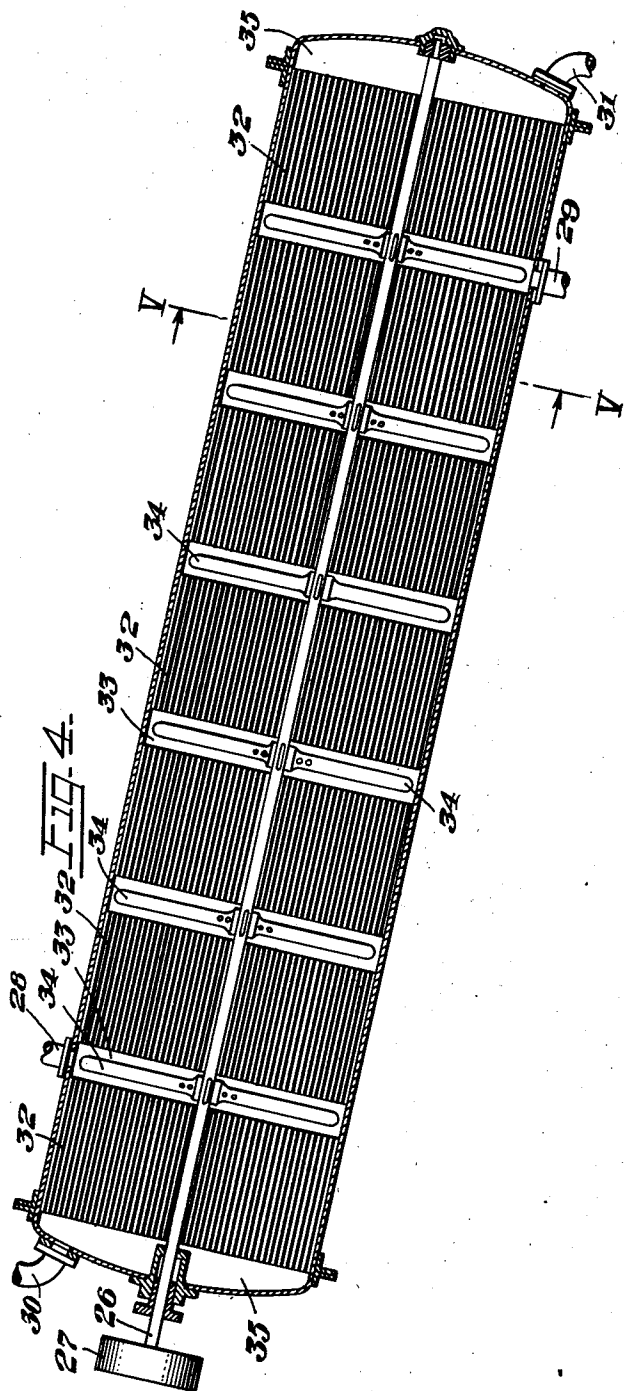
G. BOTTARO

2,218,080

PROCESS FOR COUNTERCURRENT EXTRACTION

Filed May 23, 1936

4 Sheets-Sheet 3



Inventor.
GIACOMO BOTTARO
By *Haseltine, Lake & Co.*
Attorneys

Oct. 15, 1940.

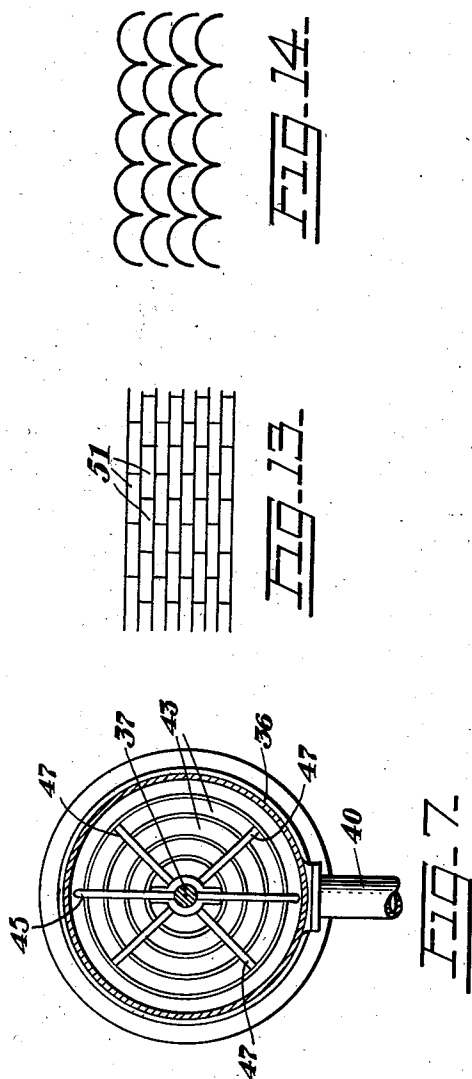
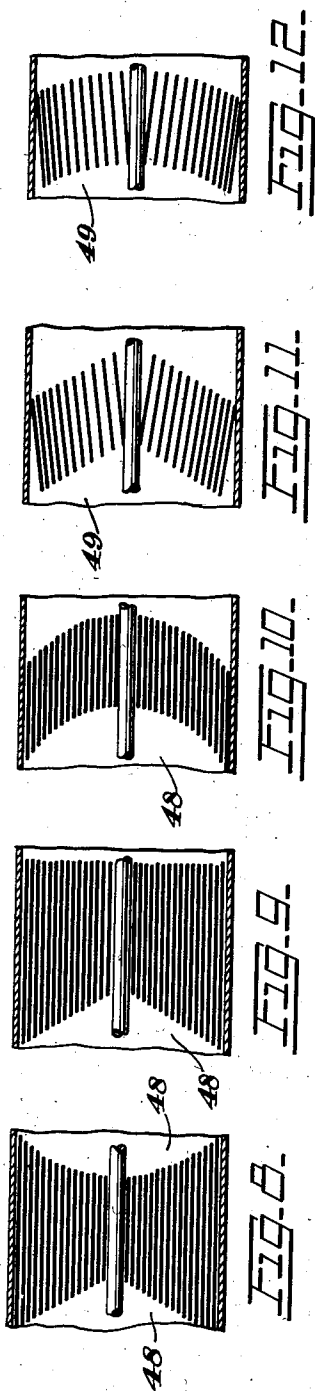
G. BOTTARO

2,218,080

PROCESS FOR COUNTERCURRENT EXTRACTION

Filed May 23, 1936

4 Sheets-Sheet 4



Inventor.
GIACOMO BOTTARO
By *Haseltine, Lake & Co.*
Attorneys

UNITED STATES PATENT OFFICE

2,218,080

PROCESS FOR COUNTERCURRENT
EXTRACTION

Giacomo Bottaro, Milan, Italy

Application May 23, 1936, Serial No. 81,378
In Italy May 25, 1935

3 Claims. (Cl. 23—268)

This invention pertains to the art of contacting countercurrently one or more liquids with one or more finely divided solids being to a great degree insoluble in the reagent liquids and differing in specific gravity and also to the art of contacting two or more liquids being to a great degree immiscible and differing in specific gravity.

Operations of this type occur very widely in physico-chemical processes as for instance in the extraction of oil from seeds, in contacting oil with bleaching earth, mineral oils with sulphuric acid, mineral oils with selective solvents, such as acetone, sulphur dioxide, furfural, propane and others for purposes of refining or dewaxing.

Apparatuses for the purpose above indicated have never been constructed before because the known mechanisms can serve only for the countercurrent contacting of liquids, but in no case have they been so constructed that their main embodiments can serve equally well for the operation of both liquid-liquid or liquid-solid systems, which feature forms the main object of the present invention.

A further object of this invention is to devise a novel construction whereby a process of countercurrent contacting may be carried out with a minimum of space in the mixing zones and a maximum of demulsifying and diffusion efficiency in the quiescence zones, so that the concentration of the extracted material in the reagent liquids may be the greatest possible while the extracted liquid or solid phases may be after contacting deprived in the greatest proportion of the materials to be extracted.

All these objects and advantages are obtained by operation in the apparatuses herein described, consisting of means forming a succession of alternate zones of turbulence and quiescence, which succession constitutes a widely used form of construction for liquid-liquid countercurrent apparatuses, which has never been employed for solids, while new constructional features are embodied. Particularly do these apparatuses all present a combination of quiescence chambers arranged in a self-contained shell and formed of a plurality of elongated straight conduits equally inclined having a constant small sectional area, extending longitudinally between two stirrer arms disposed in chambers with dimensions restricted to those strictly necessary for the accommodation of said stirrer arms, one conduit chamber being arranged at each end of said alternate succession of chambers, while outlets for the contacted phases are arranged after said terminal conduit chambers, and entrances for the phases to be con-

tacted are arranged on the chambers provided with stirrer arms. In consequence of the reduction of the space capacity in the turbulence zones, the total volume capacity is reduced to a minimum of the points in the apparatus where uniform concentration of the solute in the reagent liquids is unavoidable on account of the necessary agitation of the materials to be contacted. The result is that the highest possible concentration of the solute in the issuing reagent liquid is obtained.

The construction of the quiescence chambers or zones is characterized by the use of a plurality of straight unbaffled conduits of different shapes but all so disposed as to have an elongated form and a constant equal small sectional area which ensures that the materials pass through them in constant equal thinly laminar layers or else are divided into a large number of straight elongated streamlets allowing in both cases the separation of the phases to occur quickly due to their reduced sectional area, and thoroughly due to their elongated form, while the thinness of the laminar layers affords the greatest proportion of interfacial development of contact surface between the phases running counter-currently as against the volume of flow. Furthermore, the constant inclination of the apparatus ranging between 0° and 90° ensures the fall of the solid particles in the form of streamlets and the running of the phases in countercurrent in the quiescence zones sharply divided into two superimposed flows.

The invention completely excludes the vertical position which would destroy the effect of the division into layers of the flowing phases and in certain cases allows the horizontal position for liquids only or also for solids if the heavier phase is forwarded centrifugally. The operation is generally so conducted that the heavier liquid or solid phases are introduced in the apparatus through inlets applied on the superior turbulence zones whereas the lighter liquid phases enter through the inlets applied on the inferior turbulence zone, care being taken that an equal pressure between the liquid phases exists in all parts of the apparatus comprising inlets and outlets, so that the countercurrent movement of the phases in the quiescence zones only takes place by virtue of their different specific gravities. In a liquid-solid system, the solid phase must always be heavier specifically than the liquid phase and therefore enters the apparatus always from a superior turbulence zone.

The outlets of the reacted phases are located after the quiescence zones which terminate at

both ends the alternate succession of turbulence and quiescence chambers, while in case solid phases are to be extracted, screw conveyors are used, which do not allow the escaping liquid phase to leave the apparatus through the same outlet.

The turbulence is caused by a rotatable shaft extending through the whole length of the apparatus, carrying arms which extend in the turbulence chambers, an application which is widely known for liquid-liquid countercurrent systems but which has which has never been applied for liquid-solid systems of phases in a counter-current apparatus.

The aforesaid plurality of conduits in the quiescence zones are immovable and the separation of the phases only takes place there by the action of gravity but the invention also contemplates the application of centrifugal force to cause a quicker and more thorough separation of the phases through a shorter distance.

The apparatus in which centrifugal force is applied to can advantageously also be made to work in horizontal position, thus saving the operation of lifting the phases.

In this case the conduits assume a frustroconical or cylindrical form and they are made to rotate by fixing them on the same rotatable shaft. The heavier phase is then separated by centrifugal force and in the case of frustroconical conduits, also forwarded by it, while the lighter phase is pushed mechanically in the opposite direction of the heavier phase. The frustroconical or cylindrical conduits may have a curved re-entrance on one or both ends in order to facilitate the engagement of the phases therein.

Other details are indicated in the annexed drawings forming part hereof, and wherein several embodiments of the apparatus made according to the present invention are diagrammatically illustrated only by way of example:

Fig. 1 is a vertical axial section of a double countercurrent inclined contacting apparatus for a liquid-liquid system of phases with laminar conduits.

Fig. 2 is a sectional view taken on line II—II of Fig. 1 which shows in its upper portion a turbulence chamber and in its lower portion a quiescence chamber or zone.

Fig. 3 is a vertical axial section of an inclined countercurrent contacting apparatus for a liquid-solid system of phases with inclined laminar conduits.

Fig. 4 is a vertical axial section of a single countercurrent contacting inclined apparatus for a liquid-liquid system of phases with rotating cylindrical conduits.

Fig. 5 is a cross section on line V—V of the apparatus of Fig. 4.

Fig. 6 is a vertical axial section of a single countercurrent, horizontal contacting apparatus for a liquid-liquid system of phases with rotating frustroconical conduits.

Fig. 7 is a cross section on line VII—VII of the apparatus of Fig. 6.

Figs. 8, 9, 10, 11 and 12 are fragmentary vertical axial sections of special forms of construction of rotating cylindrical and frustroconical conduits whose ends are limited by curved surfaces.

Fig. 13 and Fig. 14 are diagrammatic partial cross sections of laminar conduits.

In several figures the design of the conduits has been only partially shown for the sake of simplicity.

Referring now to the embodiment shown in Figs. 1 and 2, 1 designates the outer shell of a cylindrical inclined container; 2 is a rotatable supported shaft extending through its whole length and provided with pulley 3; 4 and 5 designate the inlets into turbulence chambers of the heavier and lighter liquid phases to be contacted; 6 and 7 designate the outlets of the lighter and heavier phases after contacting. The numerals 8 are the laminar quiescence conduits formed by equidistant longitudinal flat plates arranged parallel to shaft 2 and extending in transverse direction over the complete chord of the cross section of the outer shell 1, while in the axial direction the ends of said plates terminate in correspondence to the cross-planes shown by the dotted lines 8a. The turbulence zones 9 are therefore determined by two reciprocally facing cross-planes 8a, which are only spaced apart by the space necessary to accommodate stirrer arms 10 fixed on a shaft 2. The conduits placed at the two ending quiescence chambers are longer than the intermediate ones. Numerals 11 designate terminal collecting chambers for the contacted liquids, while 12 are arms fixed on shell 1 for the support of shaft 2. Further means at the inlets and outlets may be used for the control of entering and leaving liquids but are not shown. Numeral 4a represents a further inlet into an intermediate turbulence chamber whenever a third phase is included in the system.

The operation of the apparatus is as follows: the lighter phase enters through 5 and fills the apparatus up to the level of outlet 6; then shaft 2 is made to rotate and the heavier phases drop through inlets 4 and 4a and displace an equal portion of the lighter phase which leaves the apparatus through 6. The heavy phase entering in 4a may be or not soluble either in the heavier phase entering through 4 or in the lighter phase entering through 5. If it is soluble in the heavier phase it will descend as a sole solution flowing countercurrently to the lighter phase since the specific gravity of said solution is higher than that of the lighter phase. If it is not soluble it will descend similarly because its own specific gravity is still higher than that of the lighter phase. If it is soluble in the lighter phase, the specific gravity of the ensuing solution must be lower than that of the heavier phase entering through 4 and then said solution rises countercurrently to said descending heavier phase.

Supposing that the heavy phase entering through 4a is soluble in the lighter phase under said conditions above and determines a sole lighter phase, the heavier phase will descend through the laminar conduits 8 and arrives in the successive turbulence zones 9 while the lighter phase ascends countercurrently through the laminar conduits 8 in the turbulence chambers 9 and from there in the above standing quiescence conduits 8 from where it proceeds to chamber 11 and outlet 6. The heavier phase continues to descend passing alternatively through all quiescence and turbulence zones till it arrives at the inferior collecting chamber 11 and leaves through outlet 7. A controlled proportionate volume of the lighter phase continually enters through inlet 5 and ascends countercurrently to the heavier phase and after dissolving the heavy phase entering through 4a it reaches outlet 6 and leaves the apparatus. The major length of the terminal quiescence conduits 8

serves to determine a complete separation of the contacted phases before leaving the apparatus.

Referring now to the embodiment shown in Fig. 3, which is particularly intended for the treatment of solids, 13 is the inclined outer shell having a rectangular shape; 14 is a rotating shaft actuated by a pulley, not shown. Numeral 15 designates the entrance into a turbulence chamber 21 of the finely divided solid phases; 16 the inlet into a turbulence chamber 21 of the liquid lighter phase; 17 the outlet of the contacted solid phase; 18 is a screw-conveyor of the contacted solid phase; 19 the outlet of the contacted lighter phase; 20 are the laminar quiescence conduits having an added inclination with the upper one of greater length in order to allow a thorough sedimentation of the finest solid particles. Said conduits are formed by equidistant longitudinal flat plates extending in a transverse direction from one side to the other of the shell 13 while in the direction perpendicular to the above, all plates begin from cross-plane 20^a and terminate alternately in cross-planes 20^b and 20^c.

The turbulence chambers 21 are therefore determined by the two mutually facing crossplanes 20^a and 20^c whose distance is the shortest required for the accommodation of arms 23; said peculiarity being omitted at the upper end of quiescence zone towards the superior chamber 24. Shaft 14 supported by arms 22 carries stirring arms 23 fixed on same, while 24 are the collecting chambers at both ends, and 13^a represent double bottoms which may be provided for the heating of the phases. Cocks 21^a serve for emptying the turbulence chambers when the apparatus is at rest, controlled exhaust openings 21^b allow gases developing from the liquid phase to escape.

The operation of this apparatus is similar to that of apparatus 1 with the sole difference that a second heavy phase is omitted and that the added inclination of the plates constituting the conduits 20 facilitate the downward course of the solid particles, and that the screw-conveyor 18 entirely stops outlet 17 so that the contacted heavier solid phase is evacuated mechanically in a manner that does not allow the lighter phase to leave through said outlet 17. The space comprised between 20^b and 20^c serves for the sedimentation of the coarser solid particles so that they will descend through the quiescence zones separately from the finer ones, thus avoiding disturbance of the course of the last ones. Means 13^a serve for heating means 21^a for the evacuation of liquid and solid materials, while 21^b serve for the evacuation of gases. These particular means 13^a, 21^a, and 21^b are only to be used when necessary.

Referring to the embodiment shown in Figs. 4 and 5, which is particularly intended when there is too little difference in the specific gravity of the phases, 25 represents the outer inclined shell; 26 the rotating shaft with a pulley 27; 28 the inlet of the heavier phase into a turbulence chamber 33; 29 the inlet of the lighter phase in another turbulence chamber 33; 30 the outlet of the contacted lighter phase; 31 the outlet of the contacted heavier phase; 32 are the cylindrical concentric laminar rotating conduits constituted by cylindrically curved plates whose ends lie in planes perpendicular to shaft 26 on which they are fixed by means of arms similar to those relating to the frustroconical embodiment illustrated in Figs. 6 and 7 and indicated by reference 47; 33 are the turbulence chambers; 34 the stir-

ring arms fixed on shaft 36; and 35 the terminal collecting chambers at both ends.

As said before this kind of apparatus can also be made to work in horizontal position.

The mode of operation of the apparatus is similar to that of the apparatus of Fig. 1, omitting a third phase, but the separation of the phases in the quiescence zones is enhanced by the centrifugal effect created by the rotation of the cylindrical conduits in the quiescence zones.

Figs. 6 and 7 illustrate an embodiment with rotating frustroconical quiescence conduits, which is particularly intended to be used when the heavier phase has a tendency to stick and hence is conveniently subjected to a forwarding action. Numeral 36 represents the outer shell and 37 the rotating shaft with pulley 38; 39 is the inlet of the heavier phase in turbulence chamber 44; 40 the inlet of the lighter phase in another turbulence chamber 44 and 41 the outlet of the contacted heavier phase; 42 the outlet of the contacted lighter phase. The frustroconical concentric laminar conduits 43 are constituted by frustroconically curved plates, fixed by means of arms 47 (Fig. 7) on shaft 37 and rotating with same; the ends of said frustroconically curved plates lie in planes which are perpendicular to shaft 37; 44 are the turbulence chambers; 45 the stirring arms fixed on shaft 37; and 46 the terminal collecting chambers at both ends.

This apparatus can also be made to work in an inclined position.

The mode of operation of this apparatus is the following: the heavy phase either solid or liquid enters at 39 and after mixing with the liquid light phase in 44 enters the frustroconical conduits 43 and overcoming the tendency to stick is forwarded centrifugally to the next turbulence chamber 44 and subsequently arrives at outlet 41. The light phase is pushed mechanically through inlet 40 and, overcoming the centrifugal force of the frustroconical rotating quiescence conduits 43, passes successively through the quiescence and turbulence zones till it leaves through outlet 42.

Figs. 8, 9 and 10 show modes of construction of the turbulence zones 33 of the embodiment of Fig. 4, by which said zones may be limited as shown at 48 in a conical or curved manner through a suitable differentiation of the lengths of the cylindrical plates forming the quiescence conduits.

Figs. 11 and 12 show modes of construction of the turbulence zones 44 of the embodiment of Figs. 6 and 7, by which said zones also may be limited in similar manner as shown at 49.

In the case of Figs. 8 to 12, the stirrer arms are preferably enlarged so as to correspond to the dimensions of the enlarged turbulence chambers. An apparatus can be formed out of a sole type of these forms of quiescence zones or of a combination thereof.

Fig. 13 is a sectional view of a type of a diaphragm type of laminar conduit to be used in connection with the embodiments shown in Figs. 1 and 3, and constituted by plates 50 extending longitudinally over the chord of the cross section of the shell of the apparatus, while the spaces resulting between two successive plates are in their turn further divided by diaphragms 51 which are interposed longitudinally between two plates and are perpendicular to the chord of said cross section. Obviously said diaphragm formation which constitutes a unitary conception for the mode of construction as shown in Figs. 1 and 3 may be substituted by sets of pipes as

equivalents, and are to be equally understood as being flat plates in the claims when such plates are specified.

Fig. 14 is a sectional view of an undulated type of laminar conduits.

Variations may, of course, be resorted to and parts used without others within the scope of the appended claims.

Having now fully described my invention, I claim:

1. In an apparatus for the continuous counter-current contacting of at least two liquid phases substantially immiscible and differing in specific gravity, as well as for the continuous counter-current contacting of at least one liquid phase with at least one substantially insoluble solid phase differing in specific gravity, while contacting the phase by superposition thereof in layers, including an alternate succession of means forming quiescence and turbulence zones with a rotatable shaft carrying stirring arms and extending through said whole succession of zones, the combination of quiescence chambers arranged in a self-contained inclined shell and formed by a plurality of elongated, clear, straight conduits having a constant equal small sectional area consisting of pipes equally inclined with the shell, extending between two stirring arms located in chambers whose dimensions are restricted to those strictly necessary for the accommodation of said stirring arms, the degree of inclination of said shell and pipes being a sufficient departure from the vertical to allow of said superposition of the phases, one conduit chamber arranged at each end of said alternate succession of chambers, outlet ports for the contacted phases arranged after said terminal conduit chambers, and entrance ports for the phases to be contacted arranged on the chambers provided with stirring arms.
2. In an apparatus for the continuous counter-current contacting of at least two liquid phases substantially immiscible and differing in specific gravity, as well as for the continuous counter-current contacting of at least one liquid phase with at least one substantially insoluble solid phase differing in specific gravity, constituted by an alternate succession of means forming quiescence and turbulence zones with a rotatable shaft carrying stirring arms and extending through said whole succession of zones, the com-

bination of quiescence chambers arranged in a self-contained shell and formed by a plurality of elongated, clear, straight conduits having a small cross-sectional area constituted by cylindrical shaped surface portions equally inclined with the shell and concentric to each other, said conduits being fixed on the rotatable shaft extending through the apparatus and arranged between two stirring arms located in chambers whose dimensions are restricted to those strictly necessary for the accommodation of said stirring arms, one conduit chamber arranged at each end of said alternate succession of chambers, outlet ports for the contacted phases arranged after said terminal conduit chambers, and entrance ports for the phases to be contacted arranged on the chambers provided with stirring arms.

3. In an apparatus for the continuous counter-current contacting of at least two liquid phases substantially immiscible and differing in specific gravity, as well as for the continuous counter-current contacting of at least one liquid phase with at least one substantially insoluble solid phase differing in specific gravity, constituted by an alternate succession of means forming quiescence and turbulence zones with a rotatable shaft carrying stirring arms and extending through said whole succession of zones, the combination of quiescence chambers arranged in a self-contained shell and formed by a plurality of elongated clear and straight conduits having a small sectional area constituted by cylindrical shaped surface members equally inclined with the shell, and concentric to each other and to the apparatus, and fixed on the rotatable shaft extending through the apparatus also while the edges of at least one of the two extremities of the several cylindrical shaped surface members lie on a curved revolution surface coaxial with the rotating shaft, said conduits being arranged between two stirring arms containing chambers whose dimensions are restricted to those strictly necessary for the accommodation of said stirring arms one conduit chamber arranged at each end of said alternate succession of chambers, outlet ports for the contacted phases arranged after said terminal conduit chambers, and entrance ports for the phases to be contacted arranged on the chambers provided with stirring arms.

GIACOMO BOTTARO.