Jan. 7, 1958 K. H. ZABRISKIE, JR 2,819,014

CENTRIFUGAL PHASE CONTACTOR

Filed Nov. 19, 1951 3 Sheets-Sheet 1

FIG. 1.

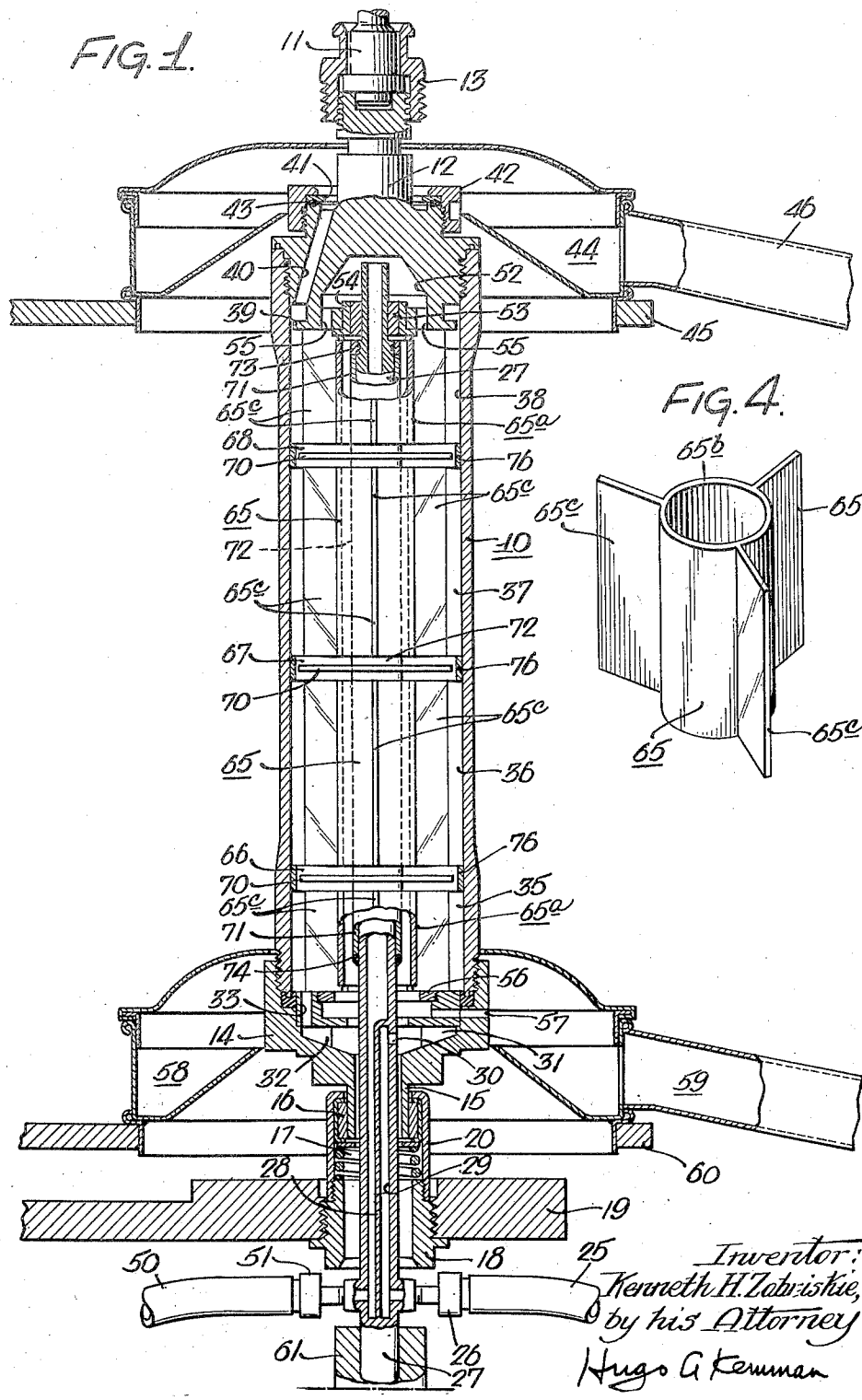


FIG. 4.

Inventor:
Kenneth H. Zabriskie, Jr.
by his Attorney
Hugo A. Kemman

Jan. 7, 1958 K. H. ZABRISKIE, JR 2,819,014

CENTRIFUGAL PHASE CONTACTOR

Filed Nov. 19, 1951 3 Sheets-Sheet 2

FIG. 2.

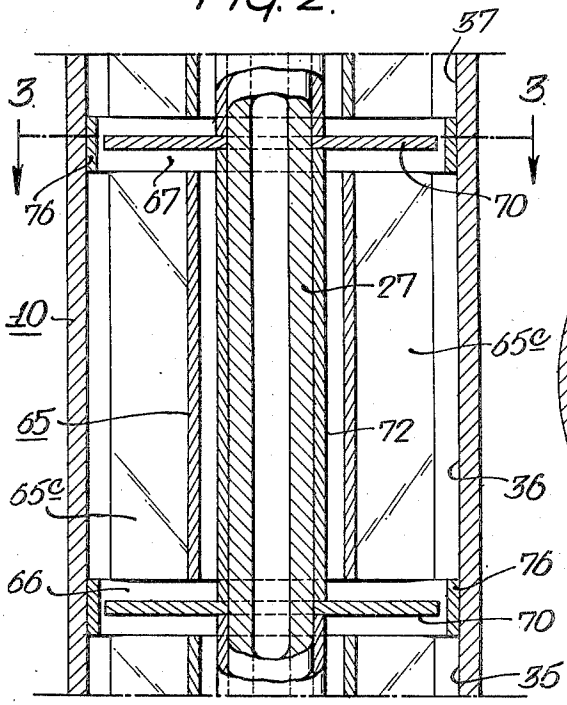


FIG. 3.

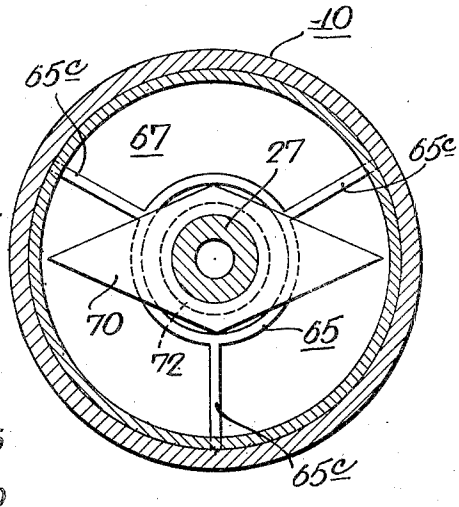


FIG. 5.

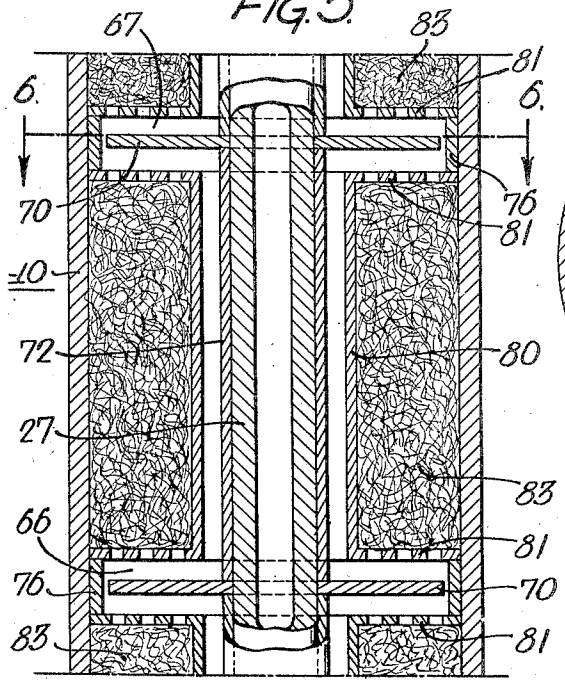
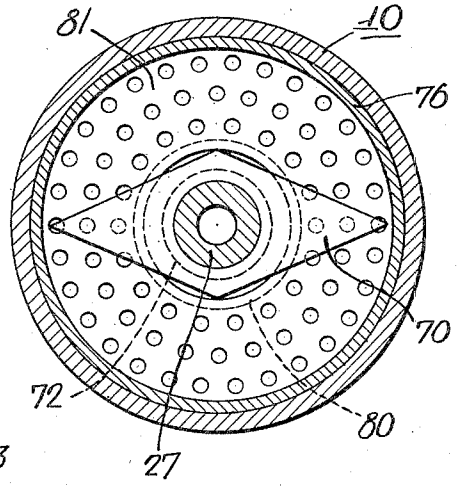


FIG. 6.



Inventor:
Kenneth H. Zabriskie, Jr.
by his Attorneys
Hugo G. Enmanuel

Jan. 7, 1958 K. H. ZABRISKIE, JR 2,819,014

CENTRIFUGAL PHASE CONTACTOR

Filed Nov. 19, 1951 3 Sheets-Sheet 3

FIG. 7.

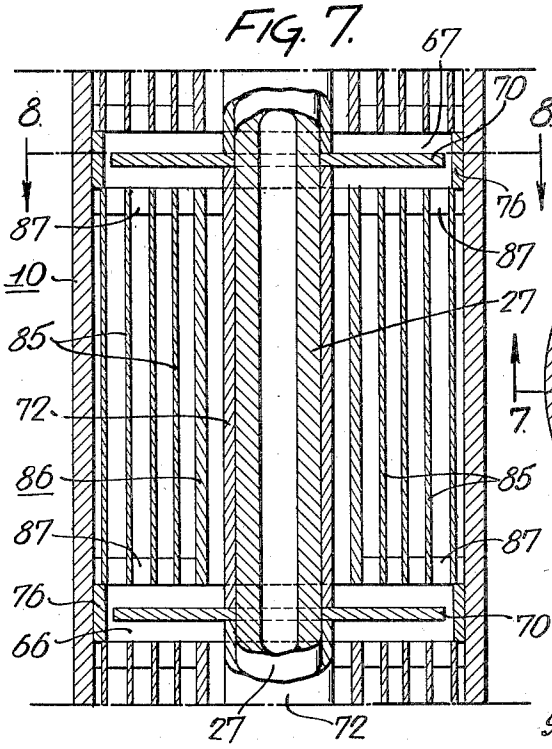


FIG. 8.

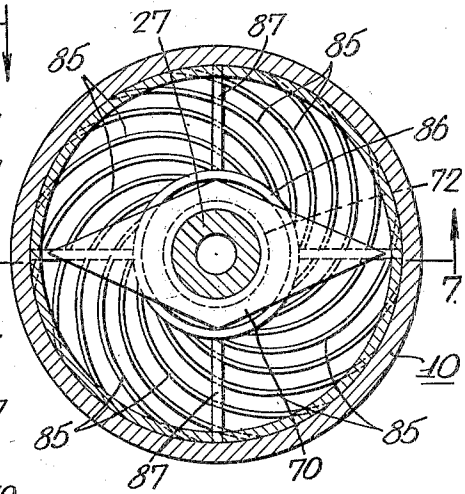


FIG. 11.

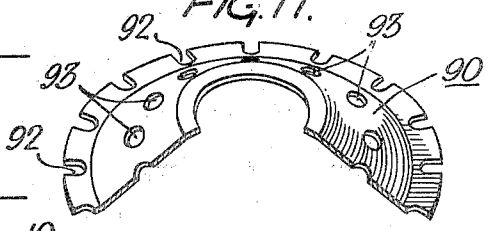


FIG. 9.

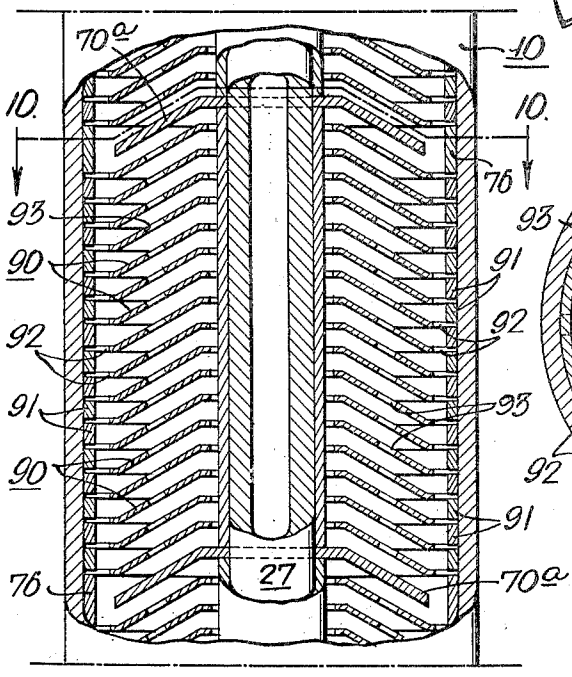
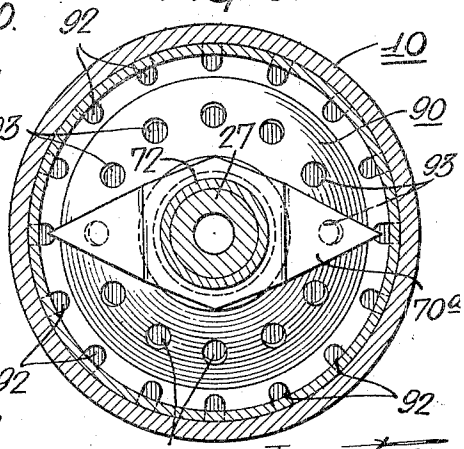


FIG. 10.



Inventor:
Kenneth H. Zabriskie, Jr.
by his Attorney
Hugo G. Fenman

United States Patent Office

2,819,014

Patented Jan. 7, 1958

2,819,014

CENTRIFUGAL PHASE CONTACTOR

Kenneth H. Zabriskie, Jr., Wilmington, Del., assignor to The Sharples Corporation, a corporation of Delaware

Application November 19, 1951, Serial No. 257,102

13 Claims. (Cl. 233—15)

This invention relates generally to a method and apparatus for the countercurrent contact of liquids, and particularly to the utilization of centrifugal force for contact and separation purposes.

Many types of equipment are now used for liquid/liquid extraction. One type comprises a series of mechanical mixers followed by gravity or centrifugal separation, each operation of mixing and settling constituting one stage, with multiple stages created by a multiplicity of mixing and separating steps, fresh solvent being introduced into the series in the stage most remote from the introduction of the feed of liquid to be extracted. Mixing may be done with some form of mechanical agitator or with pump recirculation. This system is faulty in that such mixing gives relatively poor contact efficiency. Attempts to improve contact by intensifying and prolonging the agitation frequently produce emulsions that are difficult or impossible to break.

Perhaps the most widely used device consists of a tower packed with selected material such as glass balls, Raschig rings, short cylinders, or other special ceramic shapes. The lighter liquid is introduced at the bottom of the tower and passes upwardly while the heavier liquid is fed at the top and passes countercurrently downward. Packing is ordinarily selected of a kind that one of the liquids will wet and thus comprise the dispersed phase, whereas the other comprises the continuous phase.

Other forms of extractors are well known in the art, but many of these require a great deal of floor space and/or head room, and most of them are so constructed that cleaning deposits is a major and time-consuming operation.

The principal object of this invention is to provide an improved countercurrent extraction or contact method and apparatus in which there is continuous countercurrent fluid flow through successive separating and mixing stages.

A further object of this invention is to provide an improved countercurrent centrifugal extraction or contact method and apparatus that has highly efficient contacting and separation features, and a high potential throughput per unit area.

A further object of the invention is to provide an improved centrifugal countercurrent contactor having a plurality of successive separating and mixing stages; the separating stages being relatively long with respect to the mixing stages to reduce turbulence in the central area of the separating stages.

A further object of this invention is to provide a centrifugal method and apparatus into which two liquids may be fed and through which they flow in continuous countercurrent manner, with one or more stages in which the liquids are intimately mixed, following which they are separated in each stage by centrifugal force.

Another object of the invention is to provide a more efficient countercurrent extractor that can be easily disassembled and cleaned.

Fields in which this method and apparatus are most useful include antibiotic production, including extraction from broth; extraction and purification of vitamins, hormones, aminoacids, and other pharmaceuticals; separation of close boiling-point hydrocarbons, as in the extraction of toluene or butadiene from hydrocarbon mixtures such as hydro-former effluent; extraction of oxygenated compounds from synthetic gasoline, and isomer separation of such compounds as the nitro-toluenes; recovery of organic acids such as citric, lactic, and acetic acid from aqueous solutions; purification of such organic materials as rosin, glycerine; washing of gasoline distillates to remove sulfur and mercaptans; vegetable oil fractionation; water cleanup of dilute solutions of aniline, phenol, cresol.

In order that the invention may be more readily understood, reference is made to the accompanying drawings, forming part of this specification, in which

Fig. 1 is a vertical section of a centrifugal countercurrent contactor illustrating one form of the invention;

Fig. 2 is an enlarged longitudinal sectional detail showing two mixing zones and an interposed separating zone of the device of Fig. 1;

Fig. 3 is a transverse sectional detail as seen at 3, 3 of Fig. 2;

Fig. 4 is a perspective showing the vane assemblies as used in the structures of Figs. 1–3;

Fig. 5 is a sectional detail similar to Fig. 2 in which the vanes are replaced by a fibrous material.

Fig. 6 is a transverse sectional detail as seen at 6, 6 of Fig. 5;

Fig. 7 is a sectional detail similar to Figs. 2 and 5, but showing a modified vane construction;

Fig. 8 is a transverse sectional detail as seen at 8—8 of Fig. 7;

Fig. 9 is a view corresponding to Figs. 5 and 7 showing a further modified construction for the separating stage in which a plurality of discs are used;

Fig. 10 is a transverse sectional detail as seen at 10—10 of Fig. 9; and

Fig. 11 is a perspective of one of the discs shown in Figs. 9 and 10.

The invention comprises essentially the provision in a countercurrent centrifugal contact machine of a plurality of axially spaced separating chambers or stages in the rotor. These chambers have various media for inducing or increasing separation therein and each separating chamber is adjacent to a mixing stage or chamber.

Referring now to the drawings, a centrifugal countercurrent contact machine constructed in accordance with the present invention has a rotor assembly 10 which is turned at high speed by means of a driveshaft 11 connected to the rotor top 12 by a shoulder nut 13. The rotor bottom 14 is provided with an axial extension 15 which is journaled in a bearing 16. Vertical support for bearing 16 is provided through a compression spring 17 which acts between the bearing 16 and a collar 18 mounted in the lower supporting plate 19. A cup retainer 20 for the bearing is also threaded to the collar 18.

Both the heavy and light liquids are fed from the bottom of the machine in the present embodiment. However, it will be understood that it is entirely possible to feed one or both of the liquids from the top if desired. The heavy liquid is introduced through a conduit 25 and fitting 26 to the central feed pipe 27 for the rotor. However, feed pipe 27 is provided with an axially extending baffle or wall 28 which forms a secondary feed pipe 29 for the heavy liquid. Secondary pipe 29 feeds liquid through orifice 30 into the chamber 31 which is provided with accelerating vanes 32, 32 that rapidly propel the heavy liquid outwardly and upwardly through a plurality of passages 33 into the rotor body 10. The heavy liquid then flows upwardly and through successive sepa-

rating zones or stages 35, 36, 37 and 38 from which it flows around a flange 39 on the rotor top 12 and upwardly through a plurality of passages 40, 40. It will be noted in Fig. 1 that passages 40 are tilted with respect to the center line of the rotor so that the upper or discharge ends of the passages 40, 40 are relatively closer to the center line than the lower intake ends thereof.

The depth or thickness of the heavy liquid or phase in the rotor is controlled by the inside diameter of a ring dam 41 which is retained on the rotor top 12 by a shoulder nut 42 and is provided with a gasket 43. The heavy liquid flows over ring dam 41 and nut 42 and into an annular collecting chamber 44 supported on the upper plate structure 45. Chamber 44 is provided with a suitable spout 46 from which the heavy liquid is ultimately discharged.

The light liquid is also introduced into central feed pipe 27 through conduit 50 and fitting 51 but in this case the light liquid flows upwardly and out the top of the central feed pipe 27 into a chamber 52 in the rotor top 12. The upper part of the feed pipe 27 is journaled in upper rotor bearing 53 which is retained in the rotor top 12 by means of a flanged sleeve 54. The light liquid flows into the rotor through a plurality of passages 55 which communicate with chamber 52 and the rotor.

The total depth or thickness of the heavy and light liquid layers or phases is controlled by the inside diameter of a removable ring dam 56 mounted in the rotor bottom 14. After flowing over ring dam 56 the light liquid is discharged through passages 57 into the lower collecting chamber 58 from which it is removed through the lower spout 59. The lower chamber 58 and its associated spout are supported on a plate structure 60 and the lower end of central feed pipe 27 together with its associated stationary structure may be supported by a member 61.

As previously stated, the inside diameter of ring dam 41 in rotor top 12 controls the thickness of the heavy liquid in the rotor. The total thickness of liquid, however, is controlled by the inside diameter of ring dam 56 in bottom 14 since the lighter liquid is radially nearer the axis of rotation of the rotor. The location of the dividing line between the heavy and light phases depends on the ratio of the total pressure of the light and heavy phases beneath flange 39 of rotor top 12 and the pressure of the heavy phase above flange 39 as determined by the inside diameter in ring dam 41. Increasing the inside diameter of dam 41 displaces the dividing line between the light and heavy phases outwardly in the rotor, thus reducing the thickness of the heavier layer. Decreasing the inside diameter of ring dam 41 increases the thickness of the heavy layer and similar variations in the inside diameter of ring dam 56 likewise control the thickness of the light liquid or phase and therefore the total thickness of the liquid in the rotor. The smaller the diameter the greater the total liquid thickness.

The light and heavy liquids or phases are, of course, of different density, and are at most only partially miscible.

In general, the foregoing description is directed to the structure of a countercurrent centrifugal contactor and forms no part of the present invention. In a contactor of this type, however, it becomes very important to obtain maximum efficiency and contact area between minute particles of the liquids. It has been found that satisfactory operation requires several stages in which the liquids are successively separated and mixed. The reason that several stages provide greater efficiency per stage is not completely understood but it is believed that the transfer coefficient is at a substantial maximum immediately after agitation and that continued agitation beyond a specified time interval does not materially increase transfer. However, coalescence followed by further agitation permits additional transfer. For several reasons it is desirable to incorporate the above-mentioned separating and mixing stages in a single centrifugal machine rather than utilizing separate machines for each stage. Possible disadvantages incident to placing mixing and separating zones side by side in the same rotor are overcome or minimized in the present construction with the result that additional transfer above the equilibrium value for a single stage can be achieved. The present machine provides marked increase in efficiency and reduction in cost compared to other known type of unitary countercurrent machine and compared to separate machines for each stage. The two most important factors in achieving this increased efficiency reside in the predominantly unidirectional flow of each liquid phase throughout the rotor and the ratio of the length of the separating zones with respect to the mixing zones.

In operation the heavy liquid is introduced to the bottom of rotor 10 through passages 33, 33 from whence it flows radially outward to the periphery of the first separating zone 35. This zone, together with the other separating zones, is provided with vane or wing assemblies 65 and 65*a* which materially assist the separation. Thence the heavy liquid travels upwardly through the successive mixing and separating stages or zones 66, 36, 67, 37 and 68 into the last separating zone 38.

The flow path of the light liquid from passages 55 in the rotor top is the reverse of that just described for the heavy liquid.

It will thus be understood that there is in the present machine an outer annular layer of heavy liquid continuously moving upwardly in the rotor through the separating stages and an inner layer of light liquid moving in the opposite direction through the separating stages. The direction of movement of each of these layers is not altered nor changed but in the mixing stages or zones violent agitation occurs so that the layers as such are nonexistent due to the excessive turbulence. However, the further feeding of liquid maintains the countercurrent flow in the rotor so that there is no appreciable variation in the direction thereof, nor is there appreciable variation in the angular velocity of one liquid with respect to the other, for whatever change occurs in the angular velocity of one liquid in the mixing zones is shared by the other, and since the two liquids rotate at at least substantially the same angular velocity in the separating zones, the countercurrent flow of the phases with respect to each other is under substantially the same relative angular velocity conditions.

Each of the mixing stages or phase mixing means 66, 67 and 68 comprises a stationary member or blade 70 which is mounted transversely of the rotor on the periphery of the central feed pipe or member 27 and retained thereon by a plurality of removable collars 71, 72, 72. The blades 70 and collars 71, 72 are held in place by a nut 73 which is threaded onto the feed tube 27 and compresses the blades and collars against an abutment 74 in the bottom separating stage 35. The vane assemblies 65 and 65*a* are held in place by the rotor top 12 and are axially spaced at the mixing zones 66, 67 and 68 by spacers 76, 76 which define the length of the mixing zones.

It will be understood that turbulence in and contiguous to the various phase mixing stages or zones 66–68 will be carried over to a more or less limited extent into the phase separating stages or zones 35–38. It is therefore important that the axial length of the separating stages be sufficient to provide relatively stable separating conditions in a central area of the separating stages. It will be noted that the terminal separating stages 35 and 38 are shorter than the central stages 36 and 37. Since there is a turbulent mixing zone at only one end of the terminal separating stages it is not necessary that they be so long as the central separating stages which have turbulence carried over into them from each end. Therefore the length of the terminal separating zones need only be approximately one-half the length of the central separating zones. If no vane assemblies 65, 65*a* or any equivalent devices are used in the separating stages the

length of the stages would be excessive. Therefore it has been found that turbulence and increased separating efficiency can be provided in a rotor of reasonable length if the separating zones or stages as defined generally by the axial length of the vane assemblies are at least three times the axial length of the mixing zones or stages.

From the foregoing it can be seen that the respective phases enter the mixing zones from opposite directions under conditions of countercurrent flow; that one phase is intimately dispersed in the other in said mixing zones; that said phases leave said mixing zones in admixture due to the turbulence created; and that the admixture is separated on each side of a mixing zone to maintain predominantly countercurrent flow.

Referring to Fig. 4 it will be noted that the preferred main assemblies comprise a central sleeve **65***b* to which three radially extending vanes or wings **65***c*, **65***c* are secured as by welding. When three vanes are used they are preferably spaced at 120° angles to each other. The only difference between assembly **65** and **65***a* is that the longer assemblies **65**, **65** are used in the longer central stages **36** and **37**.

Figs. 5–11 illustrate other expedients which may be used in place of the vane assembly **65** in the separating stages for the purpose of increasing separation and/or reducing turbulence therein. Fig. 5 shows the use of a separating stage assembly that comprises a central sleeve **80** which is formed integrally with or welded to a perforated disc **81** at each end. The discs and the sleeve in effect form a spool and when used in place of the vane assemblies of Fig. 4 are compressed in the rotor by top **12** and spaced from each other by collars **76**, **76**. The separating zone defined by the annular space between the discs **81**, **81** is filled with a fibrous material **83** such as glass wool.

Figs. 7 and 8 illustrate a further modified construction for the separating stages in which a plurality of spiral vanes **85**, **85** are secured to a central sleeve **86**. The vanes are radially spaced with respect to each other by means of a series of spacers **87**, **87** as shown in Fig. 8.

Figs. 9–11 illustrate a still further type of separating stage that may be found useful in which a plurality of conical perforated discs **90** are stacked in the zone. Spacing between the discs is achieved by a series of annular spacing rings **91**, **91** around the outer periphery of the discs. In this form the mixing blades **70***a*, **70***a* are bent to provide suitable clearance between discs in adjoining separating stages. It will be noted that ample circulation for the outer layer of heavy liquid is provided through the outer ring of perforations **92**, **92** and flow of the inner layer of light liquid is through the inner ring of perforations **93**, **93**.

It will be noted in all of the figures of the drawings that the various mixing blades extend through what would be the normal interface between the ligher and heavier phases if the various mixing zones were not provided. This is more clearly illustrated in Figures 9 and 10, and can be readily seen from the description of Figure 1.

It will thus be understood that I have provided an improved apparatus and process which permits substantial increase in efficiency in the countercurrent separating and contacting of liquids. The present construction permits quick disassembly of the rotor for proper cleaning which becomes highly important when the liquids tend to decompose or to leave deposits. Frequent cleaning to avoid contamination of the product is essential in certain instances.

While embodiments of the process and apparatus have been more particularly described, it is to be understood that this is by way of illustration, and that changes, omissions, additions, substitutions and/or modifications may be made by persons skilled in the art upon becoming familiar herewith, and without departing from the spirit of the invention. For instance, for purposes of illustration, the heavier phase is described as flowing upwardly through the rotor, while the ligher phase phase flows downwardly, and apparatus to effect this purpose has been particularly described. However, it will be well understood by persons skilled in the art that the apparatus may be modified in a manner to cause the heavier phase to flow downwardly while the lighter phase flows upwardly through the rotor. Then too, the rotor may revolve about an axis otherwise positioned such as horizontal, which also will be well understood. Also for purposes of illustration, with accompanying simplicity of construction, the feed pipe **27** has been described as being held stationary along with the mixing blades **70** mounted thereon. Since, broadly speaking, all that is required is relative rotational movement between the rotor and the blades **70**, it follows that such relative rotational movement may be brought about in any desired way. Thus blades **70** may be caused to rotate either in the same direction as the rotor but at a different rate, or in a direction opposite to the direction of rotation of the rotor. These variables in relative rotational movement between the mixing devices or blades and the rotor make available any desired degree of mixing or agitation, irrespective of the speed of rotation of the rotor, with increased versatility in the application of the invention. Many other modifications or embodiments will suggest themselves to persons skilled in the art upon becoming familiar herewith.

I claim:

1. A countercurrent centrifugal contactor comprising a rotor, means for feeding a liquid phase to one end of said rotor, means for feeding another liquid phase to the other end of said rotor, means at each end of said rotor for centrifugally separating said phases, means for discharging said phases in separated condition from said rotor, means for maintaining predominantly unidirectional flow of each phase through said rotor and countercurrent flow of said phases with respect to each other under substantially the same relative angular velocity conditions, and means intermediate the ends of said rotor for intimately mixing said phases while undergoing said countercurrent flow.

2. In a counter-current centrifugal contactor having a rotor, means for driving said rotor, and means for flowing partially miscible liquid phases of different density countercurrently through said rotor, the improvement which comprises means for flowing each of said phases predominantly unidirectionally through said rotor in contact with the other of said phases, means in said rotor at each end thereof for centrifugally separating said phases, means intermediate said last-mentioned means for intimately mixing said phases, said mixing means comprising a mixing member positioned transversely of said rotor, and means for effecting relative rotational movement between said mixing member and said rotor.

3. In a countercurrent centrifugal contactor having a rotor, means for driving said rotor, and means for flowing partially miscible liquid phases of different density countercurrently through said rotor, the improvement which comprises means for flowing each of said phases predominantly unidirectionally through said rotor in contact with the other of said phases, means in said rotor at each end thereof for centrifugally separating said phases, a plurality of spaced phase mixing means intermediate said last-mentioned means, each said phase mixing means comprising a mixing member positioned transversely of said rotor, means for effecting relative rotational movement between said mixing member and said rotor, and a phase separating zone positioned in said rotor between adjacent phase mixing means.

4. A countercurrent centrifugal contactor comprising a rotor, means for feeding a liquid phase to one end of said rotor, means for feeding another liquid phase to the other end of said rotor, means within said rotor for maintaining predominantly unidirectional flow of each phase

through said rotor and countercurrent flow of said phases with respect to each other under substantially the same relative angular velocity conditions, a plurality of axially spaced phase mixing zones within said rotor for intimately mixing said phases while undergoing said countercurrent flow, a plurality of axially spaced phase separating zones in said rotor, each phase mixing zone having a phase separating zone on each side thereof, and means for discharging said phases in separated condition from said rotor.

5. In a countercurrent centrifugal contactor having a rotor and means for driving said rotor, the improvement that comprises a heavy liquid inlet at one end of the rotor, a heavy liquid outlet at the opposite end of the rotor, a light liquid inlet at the same end of the rotor as the heavy liquid outlet, a light liquid outlet at the same end of the rotor as the heavy liquid inlet, means for effecting flow of each liquid from one end of the rotor to the other in a direction opposite to and in contact with the other liquid, a central member extending into the rotor, a mixing member mounted on said central member against which mixing member the heavy liquid is fed from one side and the light liquid is fed from the other side, means for effecting relative rotational movement between said rotor and said central member, and means on each side of said mixing member for separating said liquids and for maintaining predominantly unidirectional flow of each liquid during their countercurrent flow.

6. The apparatus of claim 5 wherein the means on each side of said mixing member for separating said liquids includes a vane assembly rotatable with the rotor.

7. The apparatus of claim 5 wherein the means on each side of said mixing member for separating said liquids

includes a mass of fibrous material mounted in the rotor to rotate therewith.

8. Apparatus in accordance with claim 7 in which the fibrous material is glass wool.

9. The apparatus of claim 5 wherein the means on each side of said mixing member for separating said liquids includes a plurality of conical discs mounted for rotation with said rotor.

10. The apparatus of claim 5 having a plurality of mixing members spaced axially of the central member with each mixing member having a separating zone on each side thereof for separating said liquids.

11. Apparatus in accordance with claim 10 in which the central separating zones are relatively longer than the terminal separating zones.

12. Apparatus in accordance with claim 10 in which the separating zones are provided with separating means which reduce turbulence in the separating zones, said separating means terminating in spaced axial relation on either side of the mixing members to define a mixing zone.

13. Apparatus in accordance with claim 12 in which the axial length of the separating zones is at least three times the axial length of the mixing zones.

References Cited in the file of this patent

UNITED STATES PATENTS

2,072,382	Robinson	Mar. 2, 1937
2,234,921	Webb	Mar. 11, 1941
2,705,594	Brewer	Apr. 5, 1955

FOREIGN PATENTS

659,241	Great Britain	Oct. 17, 1951