Nov. 26, 1935. G. A. KRAMER 2,022,205

APPARATUS FOR THE TREATMENT OF IMMISCIBLE LIQUIDS

Original Filed July 20, 1931 2 Sheets-Sheet 1

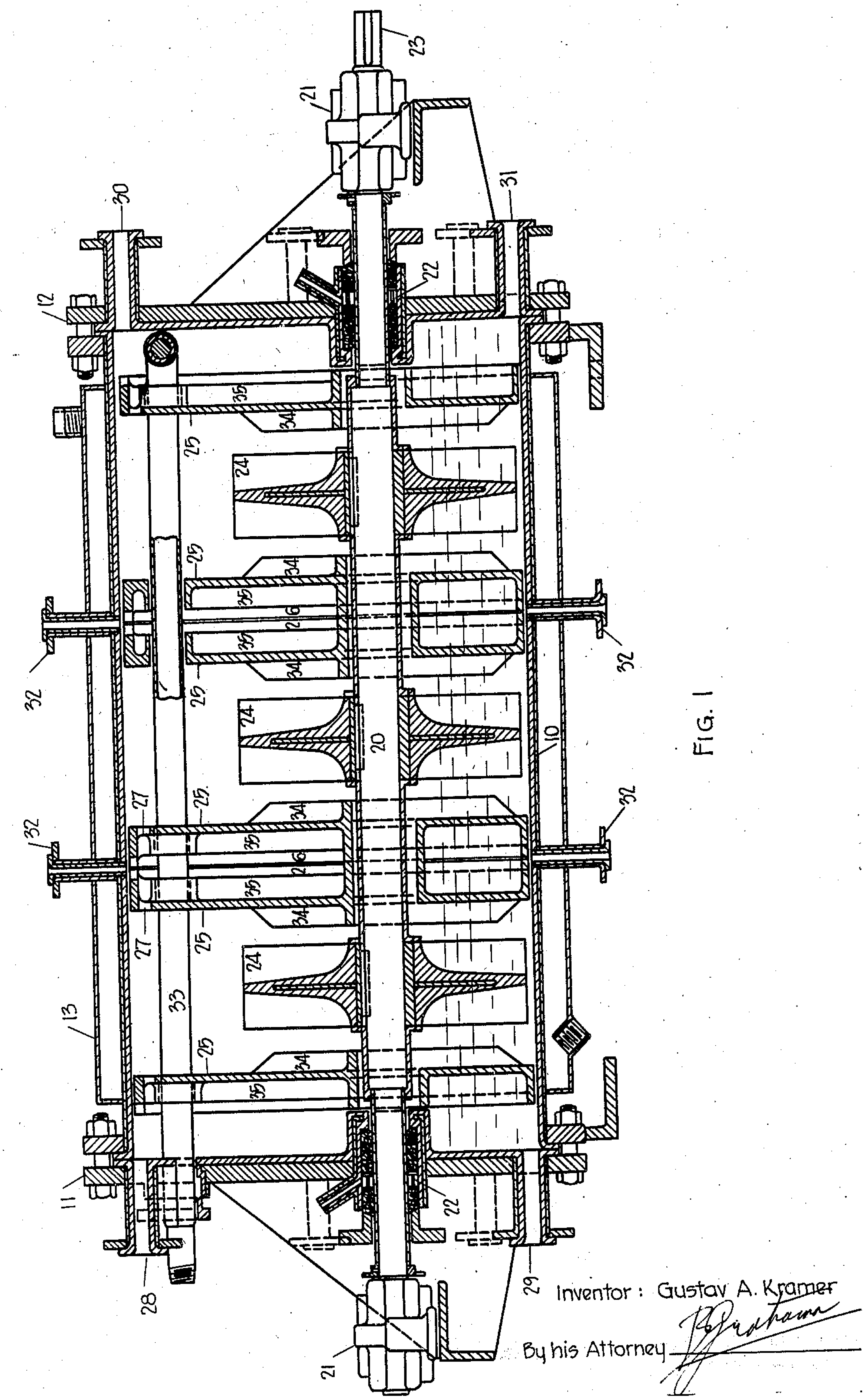

FIG. 1

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APPARATUS FOR THE TREATMENT OF IMMISCIBLE LIQUIDS

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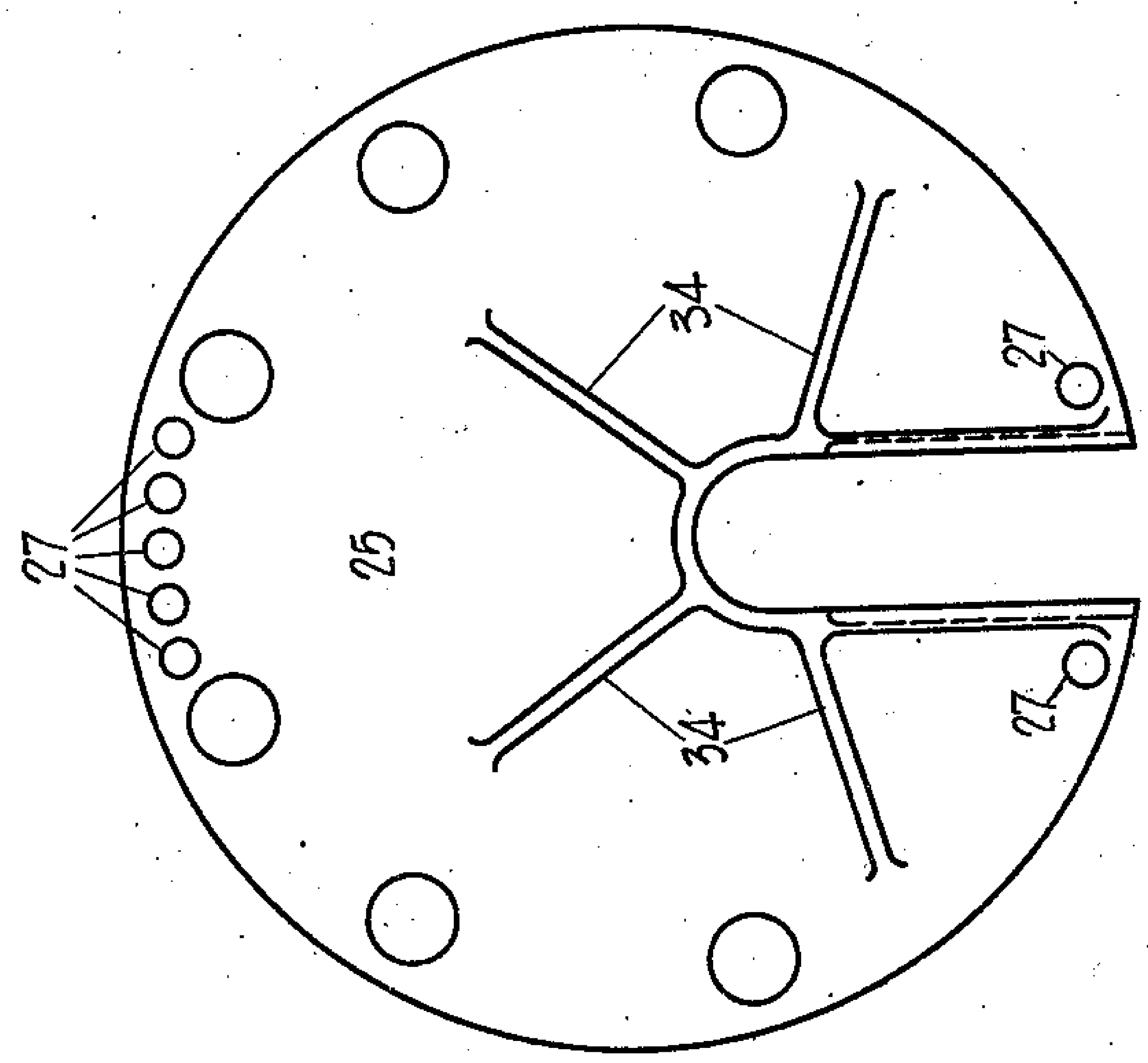

FIG. 3

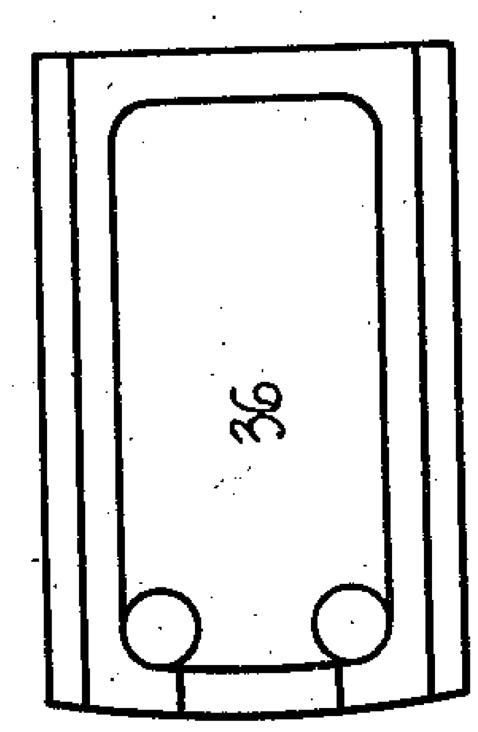

FIG. 4

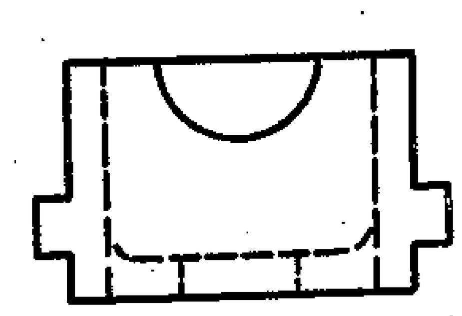

FIG. 4A.

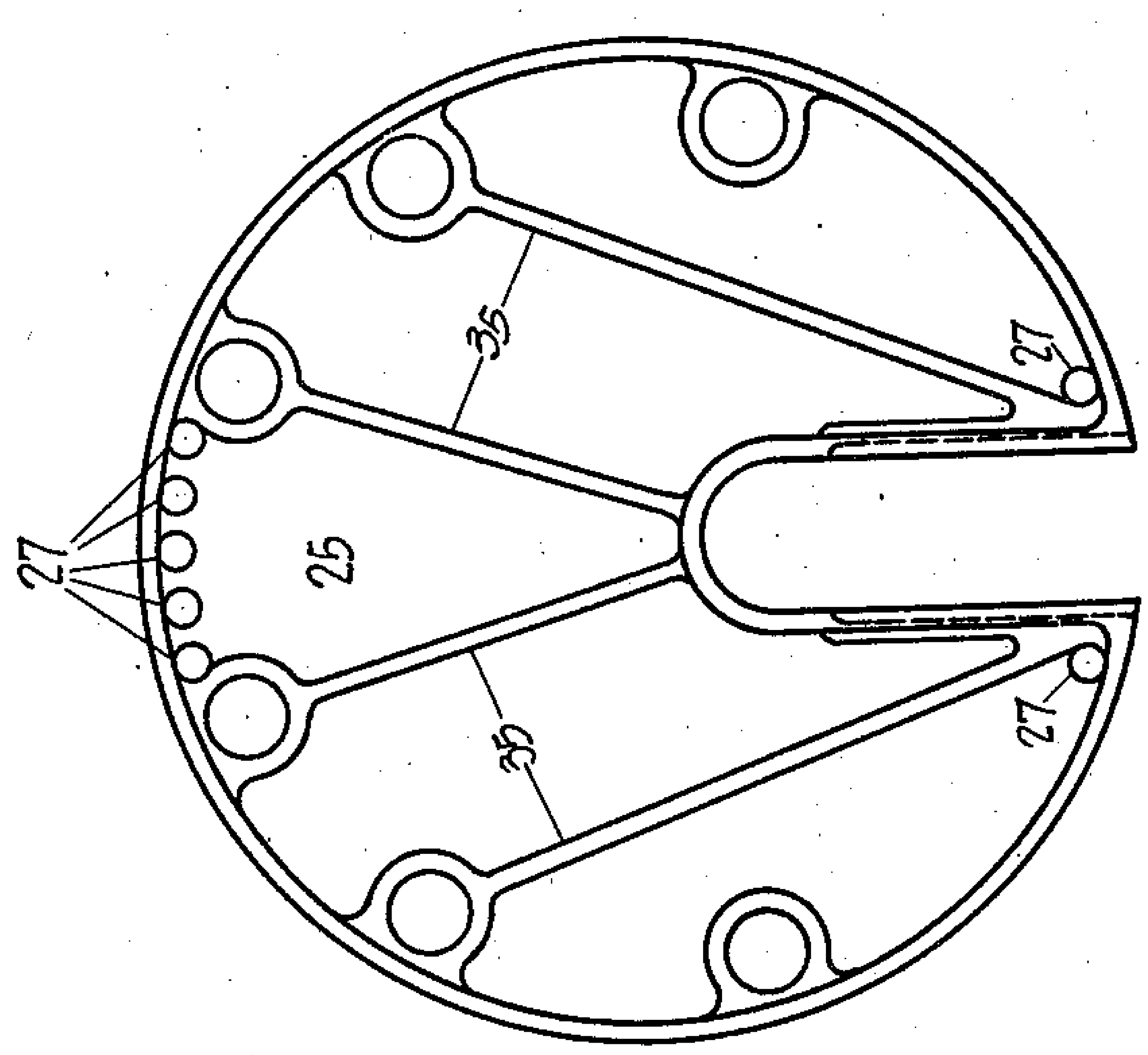

FIG. 2

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By his Attorney

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UNITED STATES PATENT OFFICE

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APPARATUS FOR THE TREATMENT OF IMMISCIBLE LIQUIDS

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Original application July 20, 1931, Serial No. 551,970. Divided and this application February 28, 1934, Serial No. 713,359. In Canada September 21, 1931

6 Claims. (Cl. 259—9)

This invention relates to a novel apparatus for contacting immiscible liquids with each other for the purpose of extraction, chemical treatment or the like, and more particularly to an apparatus wherein it is desired to carry out such contact in countercurrent relationship of the two liquids.

The preferred operation comprises passing two substantially immiscible liquids in countercurrent flow to each other through a plurality of stages arranged in contiguous relationship and communicating with each at a plurality of points, each alternate stage containing an agitating means, the end stages being characterized as quiescent or separating zones and each end stage containing an inlet and outlet for the liquids undergoing treatment. The emulsified liquids in the agitating zones are maintained in hydrostatic balance with the stratified liquids in the adjacent separating zones.

The foregoing and other objects and advantages of the invention, however, will be so clearly apparent to those skilled in the industry, as incidental to the following disclosure, that it would serve no useful purpose to further enlarge upon the same initially, and with these prefacing remarks, therefore, reference will now be immediately had to the accompanying drawings, generally illustrating at least one practical embodiment of a novel systematic combination of means for carrying forth the steps of the method involved, although not essentially the only apparatus for doing so, in which drawings

Fig. 1 is a sectional side elevation of the apparatus carried out according to this invention;

Fig. 2 is a front elevation of a diaphragm;

Fig. 3 is a rear elevation of a diaphragm;

Fig. 4 is a plan view of a diaphragm closing piece and Fig. 4a is an end view of the diaphragm closing piece.

In the drawings, wherein like characters of reference designate corresponding parts throughout the several views: 10 is a cylindrical shell provided with removable covers 11 and 12 and with an outer jacket 13 which may be utilized for heating or cooling the liquids in the mixer, should this be necessary. 20 is a shaft running parallel to the center line of shell 10 but preferably eccentrically located therein and supported in bearings 21. Stuffing glands 22 prevent leakage of liquids along the shaft when in motion, and specially formed end or ends of the shaft as shown at 23 permit connection to a prime mover by means of pulleys or couplings or the like. The shaft carries within the space enclosed by vessel 10, suitably spaced rotors 24 of any desired number which when revolving, agitate the liquid contained in the mixing device, causing intermingling of otherwise not miscible liquids in more or less fine emulsions. Diaphragms 25 are so spaced that one pair of them form a substantially enclosed space 26 in which the agitation of rotors 24 is not felt and is of such size that the emulsions formed in the mixing compartments may separate due to differences in specific gravity of the liquids in question.

Such diaphragms may be also placed between each end rotor and end plates 11 and 12 of the mixing vessel, or they may be omitted and external separating vessels substituted for this purpose.

The diaphragms 25 are closed on shaft 20 by means of diaphragm closing piece 36 which permits rotation of the shaft without substantial leakage of fluids between compartments.

The agitating and separating stages are in communication through openings 27 suitably placed near the upper and lower peripheries of the diaphragms and of such a size that they permit liquid to circulate between the two stages without causing the turbulence of the mixing zones to be communicated to the separating zones. Vanes 34 tend to increase the turbulence and eddy flow in the agitating compartments whereas vanes 35 tend to straighten out eddy flow in the separating compartments thereby expediting stratification of the immiscible fluids. Openings 28, 29, 30 and 31 are provided for feeding into and discharging from the vessel 10, the materials to be interacted, while openings 32 may be provided for sampling the liquids at various steps of the process. Coil 33 may be provided for additional internal cooling, either by water or by other refrigerating media or may be utilized for heating purposes, if so desired, by the passage therethrough of hot water, steam or other heating media.

The operation of the device may possibly be best understood by first considering the rotors at rest and supposing that two immiscible liquids of different specific gravities, e. g., sulfuric acid and hydrocarbon oil have been placed into the apparatus so that practically the entire space within the shell 10 is filled with liquid. The two liquids will naturally rest in the device in such a manner that the heavier one occupies a lower layer, the lighter one resting on top as, for example, shown by the dividing line drawn in Fig. 1.

If now the rotors are set in motion, the two liquids in the mixing compartments will be emulsified, but no substantial disturbance of the separated liquids occurs in the separating compartments because the total hydraulic head acting on openings 27 from the mixing compartment is still the same as that from the neighboring separating compartment. If we now remove some of the heavy liquid through opening 31, the deficiency will be made up by additional liquid flowing in through the openings of diaphragm 25. However, this incoming liquid contains not only sulfuric acid, but also admixed hydrocarbon, so that after separation of the two, the level of sulfuric acid in this compartment will be lower than that corresponding to the hydraulic head necessary to maintain equilibrium. Consequently, some additional liquid will flow into this separating comparment through the lower openings of the diaphragm 25 and some hydrocarbon through the upper openings of the diaphragm 25 back into the neighboring mixing compartment, until by separation of the incoming emulsion, sufficient acid has been accumulated to again establish equilibrium. Similarly, if the equilibrium were disturbed by feeding some additional acid into the opening 29, this acid will again disturb the hydraulic equilibrium and will force more acid through the lower openings of the neighboring diaphragm and so on through the entire device until equilibrium has again been reached.

By the same way, additional hydrocarbon fed into opening 30 would again be gradually distributed through the entire system, meanwhile being successively agitated with acid by the rotors in the mixing chambers.

If hydrocarbon is fed continuously into opening 30 and is being continuously removed at the same rate through opening 28, at the same time acid is being fed into nozzle 29 and being removed at the same rate through nozzle 31, it is easily understood that while the dividing levels in the separating compartments remain substantially unchanged, the acid and hydrocarbon come into thorough contact with each other in countercurrent, fresh acid always being contacted with nearly fully treated hydrocarbon and untreated hydrocarbon with nearly fully spent acid.

The relative time of contact may be varied independent of the throughput. Assume the volumetric capacity of the shell to be 200 gallons and that it contains 100 gallons of each liquid, the hourly feed also being 100 gallons of each. The average contact time of each of the liquids will then be one hour. If the contents of the shell are changed to 50 gallons of the light liquid and 150 gallons of the heavy liquid, the throughput being kept at 100 gallons per hour for each of the liquids, then the average contact time for the light liquid will be reduced to one-half hour and that for the heavy liquid increased to one and one-half hours; the ratio of the contact times of the two liquids thereby decreasing from 1 to 1/3. In similar manner, the relative time of contact of the light or heavy liquid may be either increased or decreased, depending on the character of the substantially immiscible liquids and the economic conditions of operation.

Under certain circumstances, it may be desirable to effect the contact of the two substantially immiscible fluids by means of parallel flow, in which case the lighter fluid is introduced at 30 and removed at the same rate through 28, while the heavier fluid is fed in at 31 and removed at the same rate through 29.

The number of agitating and settling chambers employed is dependent on the character of the liquids undergoing treatment and the nature of the process. For example, in parallel flow, an agitating chamber in communication with two settling chambers may suffice whereas in countercurrent flow a plurality of agitating chambers is desirable, the efficiency of the process increasing with the number of agitating compartments.

If the volumetric capacity of any of the end settling zones is insufficient to permit a well defined separation to take place, but instead an emulsified mass is present, stratification of said emulsified mass can be caused to take place by introducing said mass into one or more auxiliary surge tanks which, for all practical purposes, serve to compensate for the inadequate volumetric capacity of any end settling zone. The auxiliary tank or tanks may thus be regarded as comprising part of the end settling zone or zones.

By way of example, only, reference will be had to the treatment of a mineral oil fraction consisting essentially of hydrocarbons containing four carbon atoms to the molecule with sulfuric acid of such strength that the tertiary-base olefines contained therein, such as isobutylene, are selectively absorbed by the sulfuric acid and removed therewith, although it is to be understood that the process and apparatus is applicable to the treatment of any mineral oil fraction containing hydrocarbons of any number of carbon atoms to the molecule with an acid which may comprise H_2SO_4, H_3PO_4, HCl, etc. Pentane-pentene and hexane-hexene fractions are very suitable for treatment by my process for the selective absorption and removal of tertiary-base olefines (olefines capable of yielding tertiary alcohols upon hydrolysis).

A butane-butene fraction containing approximately 15 to 20% by weight of tertiary or gamma butylene is introduced at 30 while an equivalent amount of 65 to 70% H_2SO_4 is introduced at 29. To insure the complete absorption and removal of the gamma butylene, about a 10% excess of 65 to 70% H_2SO_4 is introduced at 29 (the amount of H_2SO_4 to be added being calculated on the amount of isobutylene to be absorbed). The vessel 10 in the meantime has been filled with 65 to 70% H_2SO_4 and the butane-butene fraction to be treated, the exact proportion of the contents of the vessel being dependent on the desired relative time of contact. The liquids flow in countercurrent fashion, the efflux at 28 comprising the butane-butene fraction from which the isobutylene has been substantially removed while the efflux at 31 comprises H_2SO_4 relatively saturated with isobutylene. The temperature is maintained between about 80° to 90° F. to avoid substantial polymerization of the butene-1 and butene-2. The pressure in the vessel is approximately the vapor pressure of the butane-butene fraction at the operating temperature.

The apparatus assures ease of control, simplicity and flexibility of operation and maximum yield in the optimum minimum time of contact.

This application is a division of my application Serial No. 551,970, filed July 20, 1931, which issued April 3, 1934, as U. S. Patent 1,953,618.

It will be obvious that various substitutions in the materials treated and in the liquids used, as well as modifications in the order and manner of execution may be made in the practical application of the invention, but such substitutions and modifications are to be considered as comprehended by the above disclosure and included within the purview of the following claims.

I claim as my invention:

1. An apparatus for the treatment of substan-

tially immiscible fluids which comprises a plurality of treating zones arranged in substantially horizontal contiguous relationship and communicating with each other at a plurality of points, each alternate zone containing agitating means, each intermediate zone comprising a settling zone and the end zones comprising separating compartments, each end zone containing an inlet and an outlet for the liquids undergoing treatment, said communicating zones being open for the free passage of fluid at a plurality of points, whose loci are at substantially opposite ends of a vertical axis drawn in the plane of a side of a zone.

2. An apparatus for the treatment of substantially immiscible fluids which comprises a vessel, a plurality of diaphragms dividing said vessel into a plurality of treating zones arranged in contiguous relationship, each of the diaphragms being perforated at the upper and lower portions of its periphery with openings of such a size that they permit liquid to circulate between two communicating treating zones without causing the turbulence of a mixing zone to be communicated to a separating zone, said lower set of perforations being so arranged that removal of fluid from an end treating zone will cause automatic flow of fluid from all the other treating zones to said end zone, and agitating means disposed in alternate treating zones, each end zone containing an inlet and outlet for the liquids undergoing treatment.

3. An apparatus for the treatment of substantially immiscible fluids which comprises a vessel, a plurality of diaphragms dividing said vessel into a plurality of treating zones arranged in contiguous relationship, each of the diaphragms being perforated at the upper and lower portions of its periphery with openings of such a size that they permit liquid to circulate between two communicating treating zones without causing the turbulence of a mixing zone to be communicated to a separating zone, said lower set of perforations being so arranged that removal of fluid from an end treating zone will cause automatic flow of fluid from all the other treating zones to said end zone, guide vanes located on the walls of the diaphragms, and agitating means disposed in alternate treating zones, each end zone containing an inlet and outlet for the liquids undergoing treatment.

4. An apparatus for the treatment of substantially immiscible fluids which comprises a vessel, a plurality of diaphragms dividing said vessel into a plurality of treating compartments arranged in contiguous relationship, each of the diaphragms being perforated at the upper and lower portions of its periphery with openings of such a size that they permit liquid to circulate between two communicating treating zones without causing the turbulence of a mixing zone to be communicated to a separating zone, said lower set of perforations being so arranged that removal of fluid from an end treating zone will cause automatic flow of fluid from all the other treating zones to said end zone, guide vanes located on the walls of the diaphragms, a shaft disposed within said vessel and eccentric with respect to the horizontal axis of said vessel, and agitating means supported on said shaft and located within alternate compartments, each end compartment containing an inlet and outlet for the liquids undergoing treatment.

5. An apparatus for the treatment of substantially immiscible fluids which comprises a series of substantially horizontally arranged agitating and settling compartments only the contiguous compartments of which are in communication with each other, agitating devices only located in alternate compartments and inlet and outlet means contained in each end compartment, said communicating compartments being open for the free passage of fluid at a plurality of points whose loci are at substantially opposite ends of a vertical axis drawn in a plane of a side of a compartment.

6. An apparatus for the treatment of substantially immiscible fluids which comprises a series of agitating and settling zones, each agitating zone being in communication with no more than two settling zones via openings of such size that they permit liquid to circulate between two zones without causing the turbulence of an agitating zone to be communicated to a settling zone, said openings being so arranged that removal of fluid from an end treating zone will cause automatic flow of fluid from all the other treating zones to said end zone, agitating devices located in alternate zones and inlet and outlet means contained in each end zone.

GUSTAV A. KRAMER.