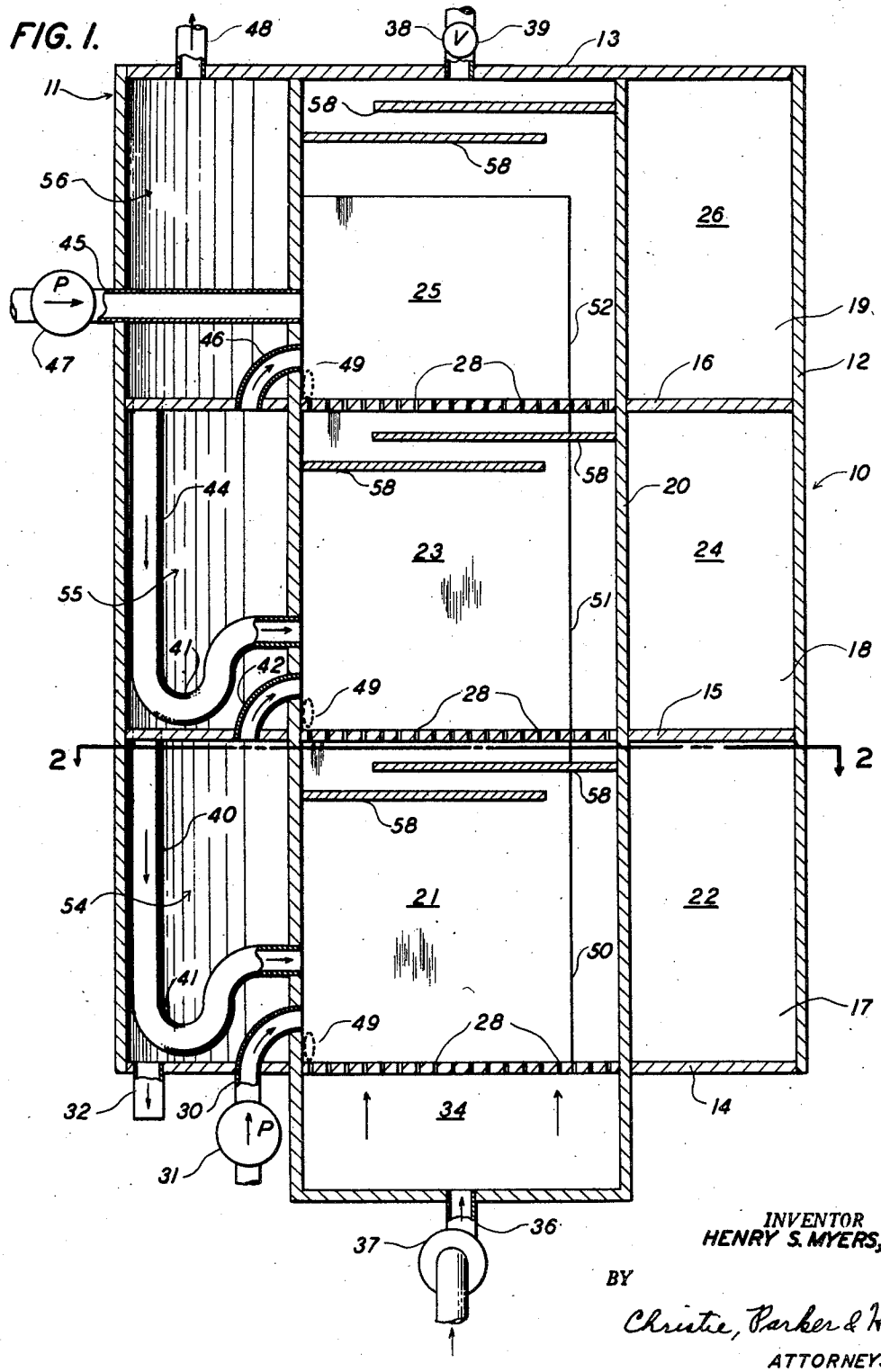
Sept. 9, 1958 H. S. MYERS, JR 2,851,396

LIQUID-LIQUID EXTRACTION

Filed Sept. 16, 1954 2 Sheets-Sheet 1

FIG. 1.



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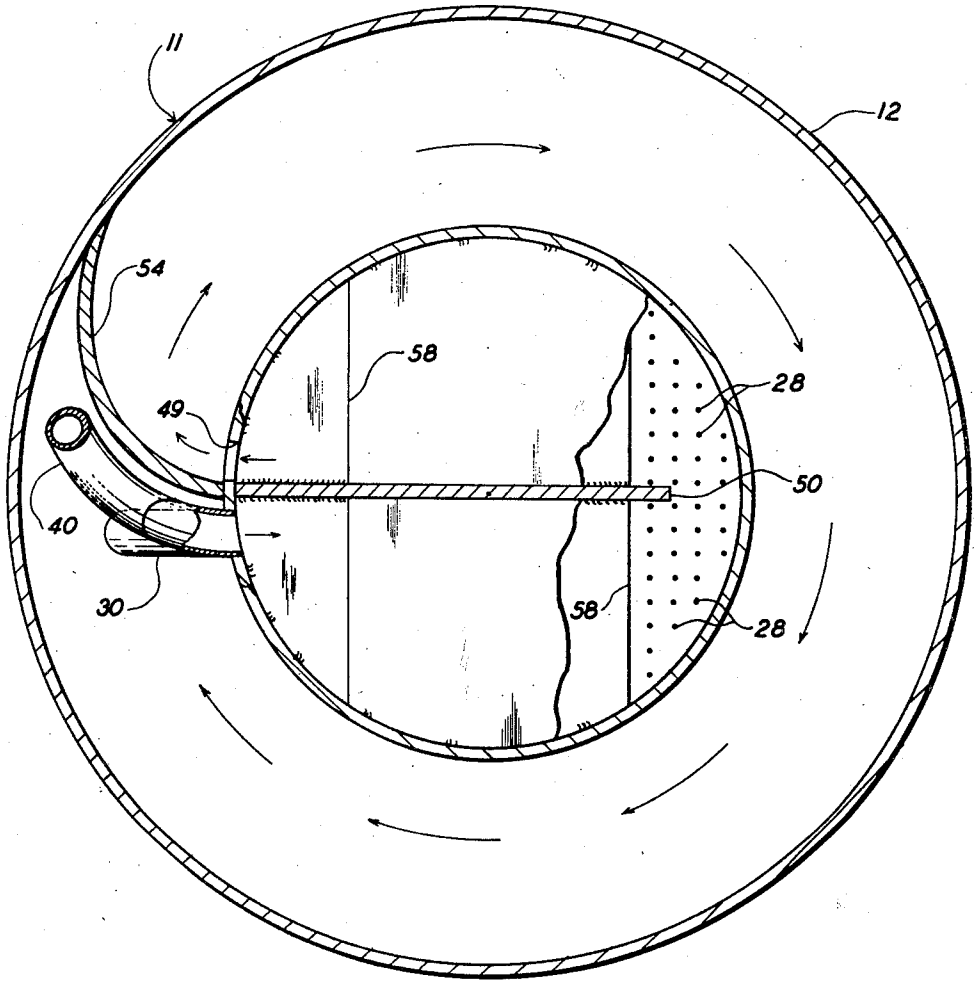
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FIG. 2.



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2,851,396

LIQUID-LIQUID EXTRACTION

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6 Claims. (Cl. 196—14.52)

This invention relates to improved apparatus and methods for liquid-liquid extraction.

Liquid-liquid extraction is a process in which a solute in a first liquid is extracted from that liquid by a second liquid intimately mixed with the first. The solute is more soluble in the second liquid than is the first liquid, and preferably the solute is more soluble in the second liquid than in the first. The two liquids are of different densities, and ordinarily they are considered mutually insoluble, although actually each one is slightly soluble in the other.

Many conventional liquid-liquid extractors use propeller type mixers in order to obtain the desired mixing of one liquid with the other. Ordinarily the propeller must be mounted in or near the interface of the two liquids and is powered by a shaft provided with conventional seals and stuffing boxes to ensure a leak-proof mixing zone. Power requirements for this type of installation are high and vibration problems are sometimes serious, often to the extent of necessitating excessively heavy and expensive apparatus.

The present invention provides methods and apparatus for liquid-liquid extraction in which propeller-type mixers and the problems accompanying such mixers are eliminated. In addition, the power requirements for conducting the process in accordance with the invention are unusually low. For the purpose of describing this invention the term "liquid-liquid extraction" is used to mean not only the operation where solute is removed from one liquid by intimate mixing with another liquid but also to include the intimate mixing of two liquids, whether a solute is present or not, to achieve a partial solution or "extraction" of each by the other. The term "solute" is used to include materials which may be suspended in the first liquid, as well as materials which are dissolved therein.

In terms of method the invention contemplates liquid-liquid extraction in which a first liquid is introduced into a mixing zone with a second liquid. The two liquids are at most only partially soluble in each other, and are of different densities. Gas is bubbled into the mixing zone to produce an intimate mixing of the two liquids. Usually one of the liquids contains a solute, and the mixing is so thorough that a substantial portion of the solute goes into the second liquid. The mixed liquids are then transferred to a settling zone where they separate.

Apparatus for carrying out the invention includes a mixing compartment and means for introducing a first liquid into the mixing zone. Means are provided for introducing a second liquid into the mixing compartment, and means are provided for bubbling gas into the mixing compartment to produce intimate mixing of the two liquids. A settling compartment is connected with the mixing compartment and means are provided for flowing the mixed liquids from the mixing compartment to the settling compartment where the two liquids separate. Means are also provided for separately withdrawing the two liquids from the settling compartment.

In the presently preferred form, the apparatus comprises an upright circular tank having a top and a bottom. The central portion of the tank bottom is perforated and an upright annular partition is disposed around the perforated portion and extends from the tank bottom to

the tank top. The partition thus divides the tank into a mixing zone or compartment overlying the perforated portion of the tank bottom and a settling zone or compartment in the annular space between the partition and the tank. A light liquid phase inlet and a heavy liquid phase inlet are provided in the partition near the tank bottom so that light and heavy phases can be introduced into the mixing zone to establish a liquid operating level therein. A mixed-liquid outlet is provided in the partition adjacent the inlets so that the mixed liquids in the mixing zone can be transferred to the settling zone. A first upright baffle is provided in the mixing zone and extends from the partition wall between the inlets and outlet inwardly so that the incoming liquids must travel around the baffle to reach the outlet. The baffle extends from the tank bottom to a height which is above the liquid operating level in the tank. A second upright baffle disposed in the settling zone extends from the partition between the outlet and inlets to the tank wall and from the tank bottom to the tank top. The settling zone is provided with a heavy liquid phase outlet and with a light liquid phase outlet. A gas chamber is disposed under the perforated portion of the tank bottom, and means are provided for introducing an inert gas into the chamber under sufficient pressure to force it through the perforations and up into the mixing zone. The top of the tank within the portion is provided with a gas outlet.

These and other aspects of the invention will be more clearly understood from the accompanying drawings in which:

Fig. 1 is a vertical section of a presently preferred form of the apparatus which may be used to conduct liquid-liquid extraction in accordance with the method of the invention; and

Fig. 2 is a view taken on line 2—2 of Fig. 1, partly broken away.

Figs. 1 and 2 illustrate an embodiment of the invention which is provided with three mixing and settling zones, but any desired number may be used.

A liquid-liquid extractor **10** comprises an upright cylindrical tank **11** having a side wall **12**, a top **13** and a bottom **14**. A lower, circular, horizontal tray **15** spaced from the tank bottom, and an upper, circular horizontal tray **16** spaced from the tank top divide the tank into lower, intermediate, and upper chambers **17**, **18** and **19**, respectively, the chambers being of equal volumes. An annular partition **20** coaxially disposed within the tank and spaced from the tank side wall extends upwardly from the tank bottom through each of the trays to the tank top to form a lower mixing zone **21** and a lower settling zone **22** immediately above the tank bottom, an intermediate mixing zone **23** and an intermediate settling zone **24** immediately above the lower tray, and an upper mixing zone **25** and an upper settling zone **26** immediately above the upper tray. Each mixing zone is within the annular partition and is surrounded by its respective annular settling zone. The portion of the tank bottom and each of the trays within the annular partition are provided with perforations **28** so that, in effect, they are sieve trays. However, other arrangements, such as bubble trays, may also be used.

A light liquid phase supply line **30** extends upwardly through the annular portion of the tank bottom outside the partition and through the partition to deliver fresh light phase to the lower mixing zone just above the perforated tank bottom. A suitable pump **31** is used to supply the light phase to the tank. A heavy phase withdrawal line **32** is provided in the annular portion of the tank bottom between the tank wall and the partition through which extracted heavy phase is withdrawn from the tank.

A gas chamber **34** is attached to the underside of the perforated tank bottom and is supplied gas through a gas inlet **36** by means of a blower **37**. A back-pressure valve **38** in a gas outlet **39** in the tank top within the annular partition controls the pressure of the gas within the upper mixing zone. The valve may be omitted if the gas is recycled, as is usually preferred.

The annular portion of the lower tray is provided with a heavy phase downcomer tube **40** which extends through the partition wall directly above the light phase supply line. The intermediate portion of the heavy phase downcomer tube is provided with a gooseneck trap **41** to prevent the light phase from passing up it. The lower tray is provided with a light phase riser tube **42**, connecting the upper portion of the lower settling zone to the lower part of the intermediate mixing zone. A heavy phase downcomer **44** is also provided in the upper tray and leading into the intermediate mixing zone directly above the riser tube **42**.

A heavy phase supply line **45** extends horizontally through the tank wall into the upper mixing zone just above a riser tube **46** connecting the upper portion of the intermediate settling zone to the lower portion of the upper mixing zone. A conventional pump **47** connected to line **45** supplies fresh heavy phase to the upper mixing zone under the desired pressure. A light phase withdrawal line **48** is provided in the annular portion of the tank top between the partition and the tank wall. Each mixing zone is provided with a mixed liquid outlet **49** (see Fig. 2) located in the partition adjacent the light and heavy phase inlets and spaced from the inlets in a clockwise direction, as viewed in Fig. 2.

The lower, intermediate, and upper mixing zones are each provided with a separate vertical mixing baffle, **50, 51, 52**, respectively. The mixing baffles in the lower and intermediate mixing zones are identically arranged. The lower mixing baffle extends vertically from the tank bottom to the lower tray and extends inwardly from the partition wall beginning between the light and heavy phase inlets and the mixed liquid outlet along a diameter of the tank to a point near the partition wall; the intermediate mixing baffle is similarly disposed within the intermediate mixing zone to extend from the lower tray to the upper tray. The upper mixing baffle is similarly arranged within the upper mixing zone except that it does not extend all the way to the tank top. The baffles in the intermediate and lower zones need not extend from tray to tray but may be stopped at any intermediate height which will be above the operating liquid levels within the respective zones.

The lower, intermediate, and upper settling zones are each provided with a separate settling baffle **54, 55, 56**, respectively. Each settling baffle begins at the partition wall between the light and heavy phase inlets and the mixed liquid outlet and extends outwardly and curves in a clockwise direction as viewed in Fig. 2 to join the tank wall. The lower settling baffle extends vertically from the tank bottom to the lower tray, the intermediate settling baffle extends vertically from the lower tray to the upper tray, and the upper settling baffle extends vertically from the upper tray to the tank top. The curvature of the settling zone baffles facilitates the smooth flow of the mixed liquids in a clockwise direction in the settling zone as indicated by the arrows in Fig. 2.

In order to reduce the tendency for liquid to be entrained from one mixing zone to the next, a plurality of horizontal baffles **58** may be installed in the upper portion of the mixing zones.

In a typical liquid-liquid extraction operation the apparatus is operated as follows: A paraffin oil containing aromatic compounds in solution is introduced into the upper mixing zone as the heavy phase through the heavy phase supply line. Liquid ammonia in which the aromatics are substantially more soluble than is the paraffin oil is introduced into the lower mixing zone through the light phase supply line. A gas, preferably an inert gas such as nitrogen or natural gas, is forced by the blower into the gas chamber and up through the perforations in the tank bottom and each of the trays. The flow of the heavy phase, light phase, and gas are in the directions indicated by the arrows in Figs. 1 and 2. The extractor may be operated with either phase continuous, and for the purpose of this explanation it is assumed that the light phase is continuous.

The gas bubbling up through the perforations in the tank bottom churns the liquid ammonia and paraffin oil supplied by downcomer **40** into a mixture of finely dispersed liquid droplets and bubbles. The intimate mixing of the two phases permits some of the aromatic remaining in the paraffin oil to pass into the liquid ammonia. The mixture passes around the end of the baffle and out the mixed liquid outlet in the lower mixing zone into the lower settling zone. In the relative quiescence of the lower settling zone the liquid ammonia and paraffin oil again separate into two phases, the heavier paraffin phase being on the bottom. The paraffin is carried from the lower settling zone through the heavy phase withdrawal line for additional extraction or other treatment as desired. The liquid ammonia continues to move upward through the riser **42** into the intermediate mixing zone and is mixed with paraffin oil coming down the downcomer **44** from the upper settling zone. The ammonia and paraffin are mixed and separate as previously described so that the ammonia passes up the riser **46** and the paraffin passes down the downcomer **40**. The ammonia then enters the upper mixing zone where it is mixed with the fresh paraffin coming in the supply line. After mixing and settling in the upper zones the ammonia containing the aromatics is taken from the tank through the light phase withdrawal line, and the paraffin from the upper zone flows down the downcomer **44** into the intermediate mixing zone. The gas which has passed serially through the lower, intermediate, and upper mixing zones is taken from the gas outlet and returned to the blower for re-use.

Under ordinary conditions, for example a temperature of about 30° C., the total pressure carried in the liquid reactor is 175 to 200 pounds per square inch. This pressure is sufficient to keep the ammonia in liquid form at that temperature. The gas pressure drop across the perforation in a particular mixing zone controls the amount of vapor space above the liquid in that mixing zone. This vapor space is necessary to prevent liquid entrainment between trays. Liquid entrainment is further reduced by the horizontal baffles provided in each mixing zone. For each mixing zone of the extractor the gas pressure drop through the perforation in the tray plus the head of liquid in the mixing zone must equal the hydrostatic head of the liquid in the corresponding settling zone. This is true regardless of the gas rate. At low gas rates the gas pressure drop is small and the depth of the liquid is high. As the gas rate is increased the gas pressure drop also increases and the head of liquid on the tray automatically falls. Thus, the tendency for liquid entrainment to occur as gas rates are increased is offset by the increase in vapor space in each of the mixing zones.

As pointed out above, the power requirements for mixing in the extractor of this invention are low, and only a single blower is required regardless of the number of trays. For a ten-stage extractor with mixing trays three feet in diameter and at eight inch vertical spacing and having $\frac{3}{16}$ inch holes on one inch triangular centers, a 30 horsepower blower is adequate. Under these conditions the inert gas is readily pumped through the extractor at the rate of about 1600 cubic feet per minute. Such an extractor has a liquid throughput of about 150 gallons per minute. At a solvent to oil ratio of two to one, this permits oil to be processed at the rate of about 50 gallons per minute.

Thus, the invention provides both methods and apparatus in which liquid-liquid extraction is accomplished by intimate mixing of two liquid phases with gas agitation which reduces vibration, and construction and operating costs. In the specific arrangement shown, maximum use is made of the space in that the liquids are caused to follow a tortuous path within the apparatus, thus obtaining long effective paths for both mixing and settling. Although in the apparatus as illustrated the mixing zone is surrounded by an annular settling zone, the arrangement can easily be reversed so that the mixing zone is annular and the settling zone is contained therein, the baffles, downcomers, risers, perforations, gas chamber, inlets and outlets being accordingly rearranged.

The method and apparatus of this invention are also useful for any operation which requires intimate contacting of two liquids even when no solute is involved. For example, it may be used to saturate one liquid with another.

I claim:

1. In a liquid-liquid extractor the combination comprising a mixing compartment having a perforated bottom and imperforate side walls, means for introducing a first liquid into the mixing compartment, means for introducing a second liquid into the mixing compartment, the second liquid being substantially insoluble in the first and of a different density, means for forcing gas up through the perforated bottom to produce an intimate mixture of the two liquids, an annular settling compartment disposed around the mixing compartment and having an imperforate inner wall surface forming a barrier preventing gas flow from the mixing compartment to the settling compartment, means for flowing the mixed liquids from the mixing compartment to the settling compartment where the two liquids separate, and means for separately withdrawing the two liquids.

2. In a liquid-liquid extractor, the combination comprising an upright tank having a wall, a bottom and a top, the central portion of the bottom being perforated, an upright imperforate partition disposed around the perforated portion to define within the tank a mixing compartment overlying the perforated portion and an annular settling compartment in the space between the partition and the tank wall, a light phase inlet into the mixing compartment, a heavy phase inlet into the mixing compartment, a mixed liquid outlet opening from the mixing compartment into the settling compartment, baffle, means for causing liquid to follow a tortuous path in the mixing compartment from the inlets to the outlet, a settling compartment heavy phase outlet, a settling compartment light phase outlet, a gas chamber underlying the perforated portion of the tank bottom, a gas outlet in the mixing compartment, and means for introducing an inert gas into the chamber the imperforate partition forming a barrier preventing gas flow from the mixing compartment to the settling compartment.

3. In a liquid-liquid extractor, the combination comprising an upright tank having a wall, a bottom and a top, the central portion of the bottom being perforated, an upright partition disposed around the perforated portion to define within the tank a mixing compartment overlying the perforated portion and an annular settling compartment in the space between the partition and the tank wall, a light phase inlet into the mixing compartment, a heavy phase inlet into the mixing compartment, a mixed liquid outlet in the mixing compartment laterally adjacent the inlets, an upright settling baffle beginning at the partition between the outlet and the inlets and extending outwardly across the settling compartment to the tank wall, a settling compartment heavy phase outlet, a settling compartment light phase outlet, a gas chamber underlying the perforated portion of the tank bottom, a gas outlet in the mixing compartment, and means for introducing an inert gas into the chamber.

4. In a liquid-liquid extractor, the combination comprising an upright tank having a wall, a bottom and a top, the central portion of the bottom being perforated, an upright imperforate partition disposed around the perforated portion to define within the tank a circular mixing compartment overlying the perforated portion and an annular settling compartment in the space between the partition and the tank wall, a light phase inlet into the mixing compartment, a heavy phase inlet into the mixing compartment adjacent the light phase inlet, a mixed liquid outlet in the mixing compartment near the inlets, an upright mixing baffle beginning at the partition between the outlet and the inlets and extending upwardly from the tank bottom and inwardly across the mixing compartment stopping short of the partition to leave a passage around the baffle, a settling compartment heavy phase outlet, a settling compartment light phase outlet, a gas outlet in the mixing compartment, and means for introducing an inert gas up through the perforations the imperforate partition forming a barrier preventing gas flow from the mixing compartment to the settling compartment.

5. In a liquid-liquid extractor, the combination comprising an upright circular tank having a wall, a bottom and a top, the central portion of the bottom being perforated, an upright annular partition disposed around the perforated portion to define within the tank a mixing compartment overlying the perforated portion and an annular settling compartment in the annular space between the partition and the tank wall, a light phase inlet in the partition near the tank bottom, a heavy phase inlet in the partition vertically above the light phase inlet, a liquid outlet in the partition adjacent the inlets, an upright mixing baffle beginning at the partition between the outlet and the inlets and extending upwardly from the tank bottom and inwardly across the mixing compartment stopping short of the partition to leave a passage around the baffle, an upright settling baffle beginning at the partition between the outlet and the inlets and extending outwardly across the settling compartment to the tank wall, a settling compartment heavy phase outlet, a settling compartment light phase outlet, a gas chamber underlying the perforated portion of the tank bottom, a gas outlet in the mixing compartment, and means for introducing an inert gas into the chamber.

6. In a liquid-liquid extractor, the combination comprising an upright tank having a wall, a bottom and a top, the central portion of the bottom being perforated, an upright partition disposed around the perforated portion and extending from the tank bottom to the tank top to define within the tank a mixing compartment overlying the perforated portion and an annular settling compartment in the space between the partition and the tank wall, a light phase inlet into the mixing compartment, a heavy phase inlet into the mixing compartment, a mixed liquid outlet in the mixing compartment laterally spaced from the inlets, an upright settling baffle beginning at the partition between the outlet and the inlets and extending from the tank bottom to the tank top and outwardly across the settling compartment to the tank wall, a settling compartment heavy phase outlet, a settling compartment light phase outlet, a gas chamber underlying the perforated portion of the tank bottom, a gas outlet in the mixing compartment, and means for introducing an inert gas into the chamber.

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