Feb. 27, 1940. G. THAYER 2,191,919

ART OF REFINING

Filed Jan. 21, 1936

Fig. 1.

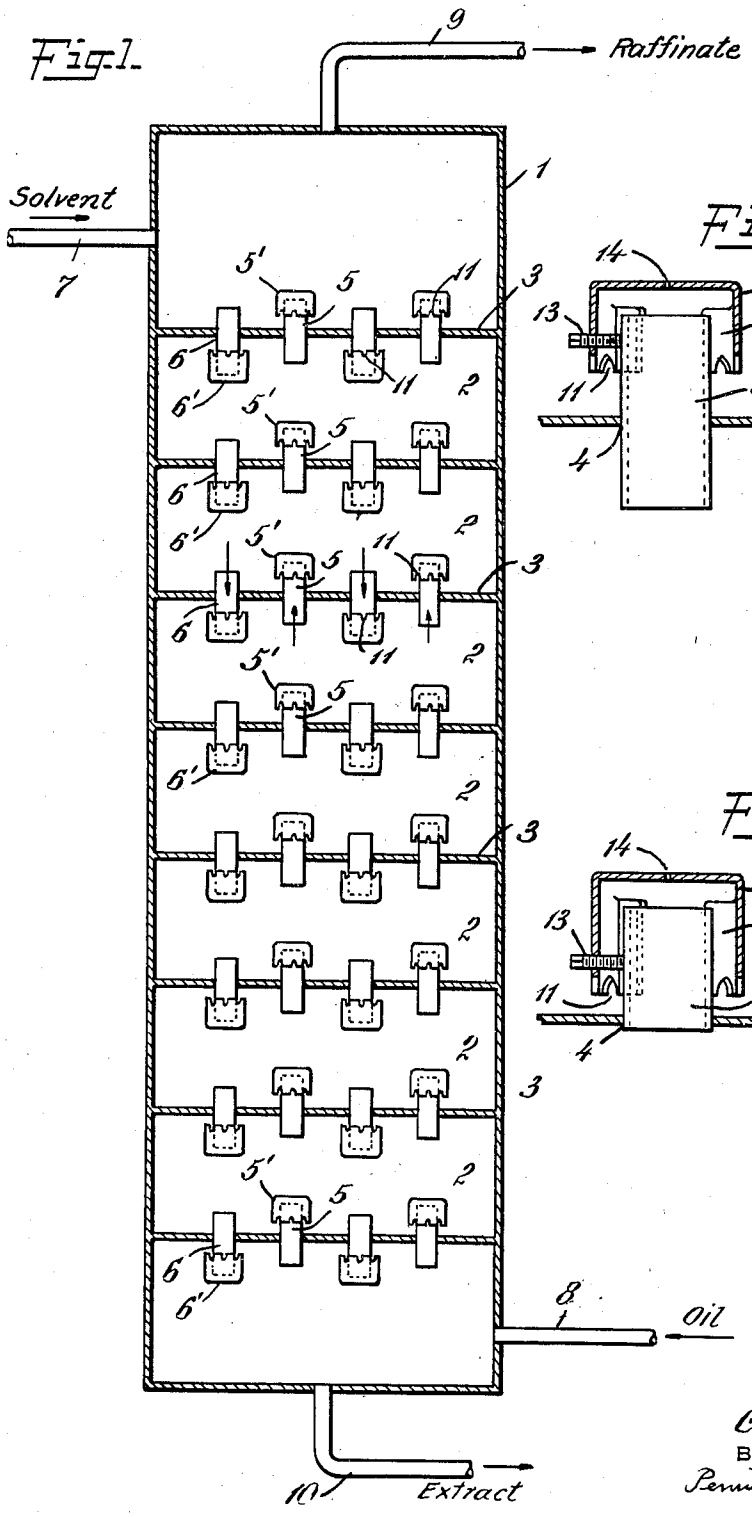


Fig. 2.

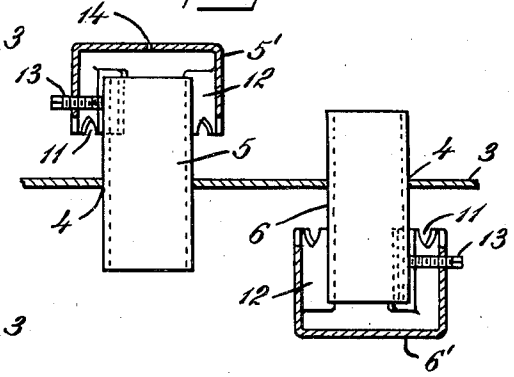
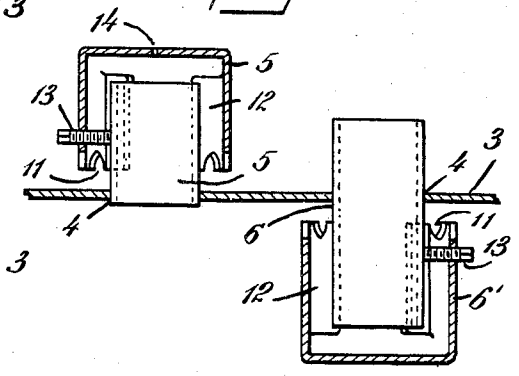


Fig. 3.



INVENTOR
George Thayer
BY
Pennie, Davis, Marvin & Edmonds
ATTORNEYS

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ART OF REFINING

George Thayer, East Chicago, Ind., assignor to Sinclair Refining Company, New York, N. Y., a corporation of Maine

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2 Claims. (Cl. 23—270)

This invention relates to improvements in apparatus for intimately contacting substantially immiscible liquids of different specific gravities in countercurrent relation. While the apparatus of the invention is generally adapted to the treatment of any two liquids of different specific gravities which are substantially immiscible in each other, it is of special utility in the refining of petroleum oils with solvents.

It is an object of the invention to provide an apparatus for treating one liquid with another substantially immiscible liquid of different specific gravity wherein each liquid will be intimately and uniformly dispersed throughout the other. Another object is to provide a contact tower in which for a tower of given size and number of compartments both the intimacy and number of contacts between the liquids will be increased.

The improved apparatus of the present invention makes use of the difference in specific gravities of the liquids to effect a countercurrent treatment of the liquids. In operation the heavier liquid will be introduced at a point in the apparatus above that at which the lighter liquid is introduced, the heavier liquid working its way downwardly and the lighter liquid working its way upwardly through the apparatus in countercurrent relation to each other. After the liquids have been contacted with each other they are withdrawn from the apparatus at points removed from their points of introduction. The apparatus of the invention comprises a housing divided into a series of superposed compartments by a series of vertically spaced horizontal partitions or trays extending transversely of the housing, means for transferring heavier liquid from the lower portion of an upper compartment and distributing it uniformly throughout the lighter liquid in the upper portion of a lower compartment, and means for transferring lighter liquid from the upper portion of a lower compartment and distributing it uniformly through the heavier liquid in the lower portion of an upper compartment. There also are provided means for introducing a liquid to an upper compartment of the series, means for introducing a lighter liquid substantially immiscible with the first-mentioned liquid to a lower compartment, means for withdrawing the first-mentioned liquid from a lower compartment than that to which it was introduced and means for withdrawing the lighter liquid from an upper compartment.

In recent years the practice of refining petroleum oils, lubricating oils particularly, with various solvents has been extensively adopted in the oil refining industry. In such solvent refining operations, naphthene base lubricating oils may, by treatment with a suitable solvent, be converted into oils having a viscosity index characteristic approximately that of paraffin base lubricating oils. Examples of solvents commonly employed for this purpose are nitrobenzene, furfural, liquid sulfur dioxide, phenol and the like. Mixtures of two or more solvents also may be used. The improvement in the properties of a lubricating oil which has been solvent refined appears to be due to the selective extraction, by the solvent, of fractions which impair certain of the properties that are desirable in a high-grade oil. The extraction treatment involves intimate contacting of oil and solvent, and for a given solvent the efficiency of the extraction depends largely on how well the oil and solvent have been dispersed through each other. The improved apparatus of the invention is of particular advantage when used in such operations as it permits the oil and solvent to be intimately and repeatedly dispersed through each other in countercurrent relation thereby promoting efficiency and economy of operation.

One generally accepted theory of liquid-liquid contact mechanics is that of a two film (for two phase systems) contact in which equilibrium exists only at the boundary. Diffusion from within the body of one of the liquids across the first film into the other film and thence into the body of the other liquid appears to be the only method of transferring the solute material. This process is accompanied by a reverse process in the other direction. In most cases of liquid-liquid contact, the diffusional rate through both films is controlling. Therefore, to improve the rate of extraction it is important that the diffusional rate for both films be increased. Solvent treating operations that heretofore have been attempted in the usual plate type treating columns have not been very satisfactory. One explanation for this is that the liquids are not being properly contacted with the result that the film surface of the liquids is small and the dispersion of one liquid through the other is inadequate, thereby permitting low diffusional rates and poor extraction. However, the apparatus of the invention provides for positive contact of each liquid with, and adequate uniform dispersion of each liquid through, layers of the other liquid. This positive contact and adequate dispersion increases the diffusional rate of the liquids thereby improving the extraction operation.

The invention will be further described with reference to the accompanying drawing in which Fig. 1 is a sectional view, in elevation, of one form of liquid contact tower embodying the invention. Fig. 2 is an enlarged fragmentary view partly in section of a part of one of the trays in the tower illustrated in Fig. 1, showing the details of one suitable arrangement of bubble caps and their relationship to chimneys or pipes extending through the openings in the trays. Fig. 3 is an enlarged fragmentary view partly in section of a part of a bubble tray suitable for use in the tower shown in Fig. 1, showing a modified arrangement of bubble cap and chimney.

The apparatus illustrated comprises a housing 1 divided into a plurality of superposed compartments 2 by horizontal partitions or trays 3 extending transversely across the housing. Openings 4 are provided in the trays to permit passage of liquid from one compartment to another. Pipes or chimneys 5 are mounted in certain of the openings 4 and extend upwardly from the trays 3. The pipes or chimneys 6 mounted in the remaining openings 4 extend downwardly below the trays 3. Bubble caps 5' are arranged in an upright position over the upper ends of the upwardly extending pipes 5. Similarly inverted bubble caps 6' are positioned over the lower ends of the downwardly extending pipes 6. The inverted bubble caps 6' extend upwardly above the lower ends of the pipes 6 a distance somewhat greater than the distance which the bubble caps 5' extend below the upper ends of the pipes 5. The exact distribution of upright and inverted bubble caps is not material so long as there is an approximately uniform distribution of the upright and inverted bubble caps, respectively, over the upper and lower surfaces of each tray. Advantageously, although not necessarily, the upwardly extending chimneys and downwardly extending chimneys also extend a short distance through the trays as illustrated in Figs. 1 and 2. In this construction the open ends of the chimneys act as weirs which insure the maintenance of layers of the heavier liquid on the upper surface of the trays surrounding the upright bubble caps and the maintenance of layers of the lighter liquid below the lower surface of the trays surrounding the inverted bubble caps, irrespective of the rate at which the two liquids are passed through the apparatus. The downwardly extending weirs may be omitted as shown in Fig. 3 if it is desired to avoid pocketing of the lighter liquid in the space immediately below the trays. Lines 7 and 8 communicate, respectively, with the upper and lower portions of the tower for supplying a heavier liquid and a lighter liquid, respectively, to the tower. Lines 9 and 10 are provided for discharging the lighter liquid and the heavier liquid, respectively, from the tower.

The particular type of bubble cap used is not material. Any of the bubble caps commonly employed in conventional bubble tray towers and which are adapted to effect a proper distribution of the material discharged around their periphery, may be used. Bubble caps conventionally used are serrated around the rim or contain peripheral slots 11 as shown in the drawing. These peripheral slots aid in dispersing the fluid passing through them uniformly through a surrounding body of liquid. It is desirable to drill a small pin hole in the upright bubble caps to avoid trapping of air gas or vapor, particularly when the tower is being started up. The bubble caps shown in the drawing are maintained in a position concentric with their corresponding chimneys by the web-like members 12. The bubble caps may be mounted on the pipes or chimneys 5 and 6 in any suitable manner. The bubble caps which are mounted in an upright position need not be attached to their corresponding chimneys. However, the bubble caps which are mounted in an inverted position advantageously are suitably attached to the lower end of the downwardly extending chimneys. One way of mounting the bubble caps on the chimneys is illustrated in Fig. 2. As there shown the bubble caps are attached to the chimneys by a screw 13 extending through the wall of the bubble cap and bearing against the side of the chimney. The number of bubble caps to be used, as well as the areas of the peripheral slots, will vary depending on the liquids being treated. In general the most advantageous ratio of the peripheral slot areas in the upright bubble caps to the peripheral slot areas in the inverted bubble caps will be determined by the ratio of the liquid quantities in the two phases. However, it also will be affected by the viscosities of the liquids.

In operation of the illustrated apparatus for the refining of a lubricating oil with a solvent of greater specific gravity than the oil, the solvent, nitrobenzene for example will be introduced into the upper portion of the tower through line 7, and the liquid of lesser specific gravity, the oil, will be introduced to the lower portion of the tower through line 8. The nitrobenzene together with the dissolved impurities, which is generally known as the "extract," due to its greater specific gravity, moves generally downwardly through the tower, entering the open ends of pipes 6 which do not have bubble caps mounted over their upper ends and thence passing through the peripheral slots 11 of the inverted bubble caps thereby being intimately dispersed through the liquid below the trays 3. Similarly the oil containing a minor quantity of the solvent dispersed therethrough, and which is generally known as the "raffinate," being of lower specific gravity than the extract and substantially immiscible therewith, moves generally upwardly through the tower, entering the open ends of the pipes 5 which are not provided with inverted bubble caps at their lower ends, and thence passing through the peripheral slots of the upright bubble caps thereby being intimately dispersed through the liquid above the trays 3. The extract and the raffinate finally leave the tower through lines 10 and 9, respectively.

In the operation just described the raffinate and extract, being substantially immiscible in each other and of different specific gravity, will tend to stratify in each of the compartments with the result that the upper part of each compartment will contain a greater concentration of the raffinate and the lower part of the compartment a greater concentration of the extract. Accordingly, that portion of the liquid, in each compartment which contains the greatest concentration of the solvent, will, upon passing downwardly into the next lower compartment of the series, be intimately dispersed through that portion of the liquid in the next lower compartment which contains the greatest concentration of the oil to be treated. Similarly that portion of the liquid in each compartment which contains the greatest concentration of the oil to be treated will, upon passing upwardly to the next higher compartment of the series, be intimately dispersed through that portion of the liquid in the

next higher compartment which contains the greatest concentration of the solvent. By repeatedly and intimately contacting those portions of the mixture containing the greatest concentration of the solvent with those portions of the mixture containing the greatest concentration of the oil to be treated, maximum treating results are obtained and the efficiency and economy of operation are improved. The upwardly extending weirs, the bubble caps and the chimneys may be made of greater length than that usually employed in conventional bubble towers if it is desired to increase the depth of the solvent layer in each compartment and the time required for the raffinate particles to pass through the solvent layers.

While the invention has been described in connection with the apparatus illustrated in the drawing it will be understood that it is not limited thereto. For example, inlet and draw-off lines may be provided at points in the tower other than the uppermost and lowermost compartments. Thus either the solvent liquid or the oil to be treated may be introduced at an intermediate point in the tower. With such an arrangement the extreme uppermost and lowermost portions of the tower would function principally as stripping sections. In general, however, the inlet and draw-off lines should be so arranged that countercurrent treatment takes place through a substantial portion of the tower.

I claim:

1. In an apparatus for the countercurrent treatment of substantially immiscible liquids of different specific gravities, the improvement which comprises a housing, a plurality of vertically spaced horizontal plates mounted within said housing and extending transversely across said housing and dividing it into superimposed compartments, a series of openings in said plates at points distributed throughout the cross-sectional area of said housing, a downwardly extending pipe secured in each of said openings in said plates for the passage of the heavier liquid in a downward direction, said downwardly extending pipes having inverted bubble caps mounted on their lower ends, a second series of openings in said plates at points distributed throughout the cross-sectional area of said housing and an upwardly extending pipe secured in each of the last-mentioned openings for the passage of the lighter liquid in an upward direction, and bubble caps mounted on the upper ends of said upwardly extending pipes, said last-mentioned bubble caps each having a pin hole in the top thereof to promote the escape of gaseous fluids.

2. In an apparatus for the countercurrent treatment of substantially immiscible liquids of different specific gravities, the improvement which comprises a housing, a plurality of vertically spaced horizontal plates mounted within said housing and extending transversely across said housing and dividing it into superimposed compartments, a series of openings in said plates at points distributed throughout the cross-sectional area of said housing, a downwardly extending pipe secured in each of said openings, in said plates for the passage of the heavier liquid in a downward direction, inverted bubble caps mounted on the lower ends of said downwardly extending pipes, a second series of openings in said plates at points distributed throughout the cross-sectional area of said housing, an upwardly extending pipe secured in each of the last-mentioned openings for the passage of the lighter liquid in an upward direction, upright bubble caps mounted on the upper ends of said upwardly extending pipes, said upright and inverted bubble caps being positioned so that the open end of the inverted bubble caps will extend above the lower end of said downwardly extending pipes a distance greater than the distance which the open ends of the upright bubble caps extend below the upper end of said upwardly extending pipes.

GEORGE THAYER.