

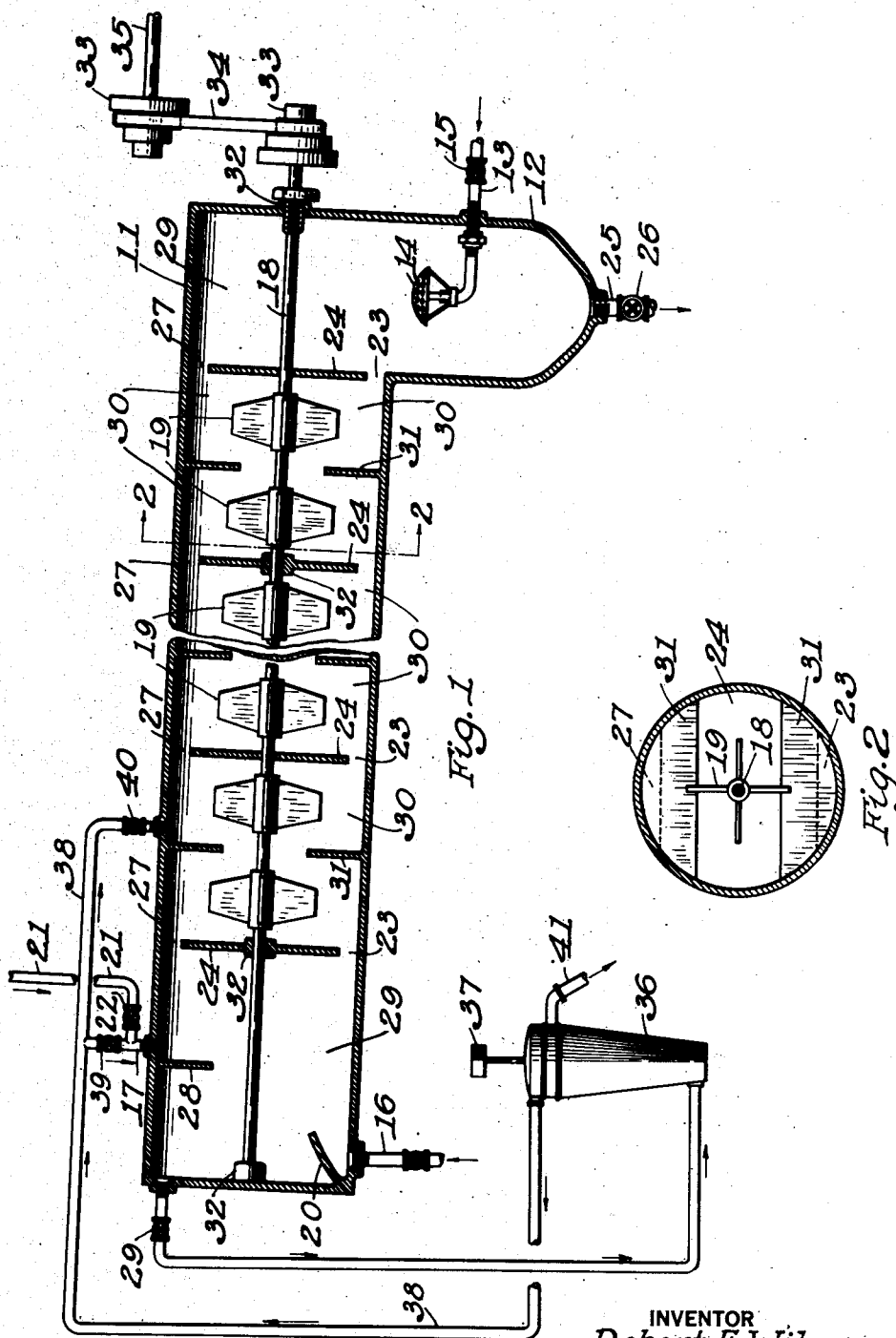
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PROCESS AND APPARATUS FOR CONTACTING TWO MATERIALS

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PROCESS AND APPARATUS FOR CONTACT-
ING TWO MATERIALSRobert E. Wilson, Chicago, Ill., assignor to Stand-
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6 Claims. (Cl. 196—13)

My invention relates to processes and apparatus for the continuous counter-current contacting of two materials, particularly two substantially liquid materials. More particularly it relates to processes and apparatus for the continuous counter-current extraction of a mineral lubricating oil stock with a selective solvent.

It is an object of my invention to provide a more efficient, effective and economical process and apparatus for continuous counter-current contacting of this type than any heretofore available. In particular it is my object to provide a novel and advantageous type of process and apparatus for the continuous counter-current extraction of a mineral lubricating oil stock with a selective solvent. The various detailed objects of my invention will become apparent as the description thereof proceeds.

Mineral lubricating oil stocks, particularly "mixed base" stocks, as produced by distillation processes from crude mineral oils contain a great variety of chemical constituents most of which have never been individually identified but which may be roughly classified as "relatively paraffinic" constituents and "relatively naphthenic" constituents. I refer to any fraction of the original lubricating oil stock which has properties nearer those of paraffinic hydrocarbons than are the properties of the original stock as a "relatively paraffinic fraction." The segregation of a "relatively paraffinic fraction", for example by solvent extraction, leaves a fraction of the original stock having relatively non-paraffinic properties. This latter fraction is composed of "relatively naphthenic constituents" and will be referred to herein as a "relatively naphthenic fraction." In general, the relatively paraffinic fraction can be identified as having a high ratio of hydrogen to carbon, a low temperature coefficient of viscosity, and a high resistance to sludging as compared with the relatively naphthenic fraction.

It is well known that the relatively paraffinic constituents are preferable to either the relatively naphthenic constituents or the original mineral lubricating oil stock for use as an ordinary lubricant. For this reason many attempts have been made to separate these relatively paraffinic constituents from the accompanying relatively naphthenic constituents in order to produce a superior lubricating oil. In the past this has frequently been done by extracting the mineral lubricating oil stock with a selective solvent for either the relatively paraffinic constituents or the relatively naphthenic constituents, generally the latter.

The principal procedures which have been used in this type of process to date have been:

1. More or less completely dissolving the lubricating oil stock in a solvent at an elevated temperature and then lowering the temperature so as to throw a relatively paraffinic fraction out of solution as a separate liquid phase; and

2. Agitating the mineral lubricating oil stock with a solvent in a tank or similar vessel at such a temperature that a fraction (generally the relatively naphthenic fraction) is dissolved and the remaining fraction is left as a separate liquid phase.

The process and apparatus of my invention improve on these prior art practices by passing the mineral lubricating oil stock and a selective solvent in counter-current relationship to each other and in continuous or preferably intermittent contact with each other, coagitating them throughout an intermediate portion of their contact zone and removing the resultant materials, which are, in general: (1) the bulk of the selective solvent carrying with it in solution a relatively naphthenic fraction of the original mineral lubricating oil stock, and (2) the residual relatively paraffinic fraction carrying with it some dissolved solvent and generally some entrained material. My process has the advantage of continuity, requires very little equipment for a plan of large capacity, is very cheap as compared with former processes, is much more flexible and controllable than the prior art batch processes and accomplishes a more efficient extraction, i. e., there is more divergence in properties between the relatively paraffinic and the relatively naphthenic fractions as I produce them than between the corresponding fractions as produced by the prior art processes with which I am familiar.

Reference will now be had more particularly to the drawing in which: Figure 1 represents a longitudinal view of my apparatus, partly in section, and Figure 2 represents a partial transverse section drawn along the line 2—2 of Figure 1.

The central feature of my apparatus is a generally horizontal elongated chamber 11 carrying a downwardly elongated settling chamber or sump 12 at one end and equipped with various baffles, paddles and other fittings which will be described later. An upwardly elongated settling chamber similar to sump 12 may, if desired, be used at the end of chamber 11 opposite sump 12 and the pipe controlled by valve 29 may suitably lead off from a point near the top of said up-

wardly elongated settling chamber. Although chamber 11 should be generally horizontal, I prefer to tilt it at a slight angle from the horizontal, as indicated in the drawing. Chamber 11 may be of any suitable cross section, for instance circular (as shown), U-shaped or elliptical with the vertical axis longer than the horizontal. At the end of the chamber 11, which carries the sump 12, a pipe 13 enters the chamber and is upturned to permit the introduction of a fluid material to chamber 11 in an upwardly-directed stream. The upturned end of pipe 13 is shown as carrying a perforated baffle 14 adapted to distribute the incoming material. At the opposite end of chamber 11 a pipe 16 enters the chamber at the bottom thereof and a pipe 17 enters at the top, one or both of which are used for the introduction of the second material to be contacted.

As a specific embodiment of my invention, I utilize my process and apparatus to extract a mineral lubricating oil stock, namely, a wax-bearing "cut" made by distillation of a Mid-Continent crude oil and having a viscosity of 74 seconds Saybolt at 210° F., with a selective solvent, namely, dichlorethylether. Since the solvent has a higher specific gravity than the lubricating oil stock it flows through my apparatus as a lower stratum and the lubricating oil stock flows in the opposite direction as an upper stratum.

If my process is operated without rotation of shaft 18 carrying paddles 19 the operation is as follows: The dichlorethylether is introduced through pipe 16, is deflected by baffle 20 and passes along the bottom of chamber 11 through passages 23 which are formed by chamber 11 and baffles 24. Dichlorethylether may also, or alternatively, be admitted through pipe 17 by means of pipe 21 and valve 22 and if so it flows downwardly through the outgoing stream of extracted lubricating oil stock, thereby washing out at least a portion of the entrained "dirty" dichlorethylether carried by said extracted lubricating oil stock, passing to the bottom of chamber 11 and flowing through passages 23 in succession along with the dichlorethylether, if any, admitted through pipe 16. The dichlorethylether (carrying extracted relatively naphthenic constituents of the original lubricating oil stock) flows out through the last passage 23 on the right and comes in contact with the incoming lubricating oil stock introduced through pipe 13. This is desirable since this incoming stock is often highly viscous and by allowing it to dissolve a portion of the solvent its viscosity is reduced and it flows through the apparatus more readily than would otherwise be the case. The "spent" dichlorethylether then passes into sump 12 which is elongated to permit entrained undissolved oil to settle out and the settled dichlorethylether with the dissolved relatively naphthenic fraction is removed from sump 12 through pipe 25 and valve 26. The material removed through pipe 25 is then subjected to a fractional distillation operation to remove the dichlorethylether from the relatively naphthenic fraction of the lubricating oil stock dissolved therein. The dichlorethylether, after purification, if desired, is then returned to the process through pipe 16 and/or pipe 17 and the relatively naphthenic fraction is available as a by-product.

Simultaneously with the introduction of dichlorethylether through pipe 16 and/or pipe 17, lubricating oil stock is introduced through pipe

13, preferably at about one-half the rate at which the dichlorethylether is introduced. The lubricating oil stock, after picking up some of the spent dichlorethylether as previously described, flows upwardly through compartment 28 and thence in succession through passages 27 formed by chamber 11 and baffles 24. Flowing out of the last passage 27 on the left it comes in contact with fresh dichlorethylether introduced through pipe 17, passes under baffle 28 (which is used to preserve quiescence in the vicinity of the outlet) and is continuously removed from chamber 11 through valve 29.

Baffles 24 in their preferred form extend transversely across chamber 11 and serve to divide it into a series of compartments including end compartments 29 and intermediate compartments 30. As hereinbefore mentioned, baffles 24 do not extend to the top or bottom of chamber 11 but are cut off to form segmental passages 23 and 27 at the bottom and top respectively. In other suitable forms of my apparatus, these passages may be formed by openings located near the top and bottom of baffles 24 and may be staggered so that passages in adjacent baffles are not in alignment and material flowing through them must therefore pursue a circuitous course. When working with highly viscous materials, it is sometimes desirable to use circular passages and to place a small screw conveyor in each passage, or in certain of them to expedite the flow of material.

In the center of each compartment 30 are located two secondary baffles 31 extending downwardly from the top of chamber 11 and upwardly from the bottom thereof, respectively. These baffles 31 are preferably circular segments as shown and it is preferred that they extend to points considerably closer to the axis of chamber 11 than do passages 23 and 27. The purpose of baffles 31 is to force the materials flowing through chamber 11 to pass into the central portion of chamber 11 at intervals throughout their flow.

Extending throughout the length of chamber 11 and approximately axially disposed therein is shaft 18 which is carried by bearings 32 as shown. Shaft 18 is driven through pulley wheels 33 and belt 34 by means of drive shaft 35 which is rotated by means not shown. The speed of rotation of shaft 18 can be varied at will by changing the position of belt 34 on pulley wheels 33. Any suitable means for selectively adjusting the speed of rotation of shaft 35 can be used but it is highly important that some such means be provided.

Within each half of each of compartments 30, shaft 18 carries a paddle 19 suitably mounted to rotate with shaft 18. Paddles 19, as shown, are composed of four flat blades slightly tapered so as to give maximum agitation at the center and slightly reduced agitation at points progressively nearer to the periphery of chamber 11. It is preferred that the blades of paddles 19, when in vertical position, extend somewhat closer to the top and bottom of chamber 11 than do the inner edges of baffles 31 but not as close as do the outer edges of baffles 24.

Various modifications can be made in paddles 19. In some cases it is desirable to perforate them so as to obtain an increased emulsifying effect. A still higher degree of agitation can, when desired, be obtained by using a greater number of blades on each wheel and/or by making the blades cup-shaped rather than flat.

Having now described the principal characteristics of my process and apparatus with the ex-

ception of the centrifugal separation feature to be described later, I will turn now to a more detailed discussion of the flow of materials through chamber 11. In the specific embodiment described above I have indicated the flow of materials when no agitation is used, i. e., when shaft 18 and paddles 19 are stationary. Under such conditions, however, there is little effective extraction because the contact between the dichlorethylether and the lubricating oil stock is entirely inadequate. By rotating paddles 19 at a moderate speed, generally within the range 8 to 50 R. P. M., it is possible to create a well mixed emulsified stratum in the central part of the apparatus, quite adequate to give effective contact, and yet by having the paddles fairly short, as shown, continuous separation of the two materials in each of compartments 30 can be obtained. The result of operating under such conditions is that there is always a stratum of substantially unemulsified lubricating oil stock at the top of each of the compartments, a stratum of substantially unemulsified dichlorethylether at the bottom of each of the compartments and an intermediate well-mixed or emulsified layer. As aforementioned, the upper limit of the emulsified layer should be above the lower edges of the upper baffles 31 and below the upper edges of baffles 24 and the corresponding lower limit should be below the upper edges of baffles 31 and above the lower edges of the lower baffles 24. By adjusting the speed of rotation of shaft 18 in order to achieve this result it is possible to prevent any substantially emulsified material from passing from one compartment to the next and to force the lubricating oil stock and the dichlorethylether to pass into the emulsified stratum in each of compartments 30, thereby insuring that the two materials come into intimate contact in each of compartments 30 before again settling out and passing on to the next compartment. In operating as described, I find that I can obtain substantially complete equilibrium contacting of lubricating oil stock and dichlorethylether in each contact zone of my process (i. e., in each of compartments 30) by so adjusting the rate of introduction of the starting materials as to make the time of contacting in each stage about two to four minutes. By using 5 or 6 stages I obtain an extremely efficient countercurrent extraction such as could be secured only by a great many separate and time-consuming steps when using the old prior art processes.

The rate of rotation of shaft 18 which will accomplish this desired optimum degree of emulsification will vary greatly with such factors as the character of the materials being contacted, the temperature, the design of the paddles, etc. but in typical cases it will be within the range 5 to 60 R. P. M.

My apparatus can be designed to have any number of compartments 30 from one up. However, in order to take full advantage of the counter-current principle, I prefer to use at least three compartments 30, and preferably four or five of them in addition to the two end compartments 29. In general it is not desirable to use more than six compartments since the advantage gained by the addition of further compartments is not sufficient to offset the additional cost.

A further feature of my process and apparatus, which has not heretofore been discussed, is a method for removing entrained material from one or both of the resultant materials withdrawn from chamber 11. The drawing shows this re-

moval of entrained material as operating on the lighter of the two resultant materials leaving chamber 11. In the specific embodiment heretofore referred to, this lighter material is the extracted lubricating oil stock, i. e., the relatively paraffinic fraction together with some dissolved dichlorethylether and some entrained material. Rather than subject this whole system, as withdrawn from chamber 11, to distillation to separate dichlorethylether from oil as would normally be done, I prefer to first remove the entrained material since this is composed for the most part of "dirty" dichlorethylether carrying some of the relatively naphthenic constituents of the original lubricating oil stock which it is desired to segregate from the relatively paraffinic constituents constituting the bulk of the material removed through valve 29. According to my process, this separation of entrained material is done by the use of centrifuge 36 operated through pulley 37 by means of a motive source not shown. The relatively paraffinic fraction produced in chamber 11 carrying some dissolved solvent and some entrained "dirty" solvent is withdrawn from casing 11 through valve 29 and passed to centrifuge 36 where the entrained material is separated from the relatively paraffinic fraction containing dissolved solvent and said entrained material is returned by means of pipe 38, and, if necessary, a suitable pump not shown, to casing 11 through valve 39 and/or valve 40. The relatively paraffinic fraction carrying dissolved solvent passes out of centrifuge 36 through pipe 41 and is then, in general, subjected to fractional distillation, by means not shown, to separate the dichlorethylether from the relatively paraffinic fraction which constitutes the principal final product of my process. The dichlorethylether removed from this relatively paraffinic fraction, after purification if required, is returned to the process by means of pipe 21 along with the solvent recovered from the relatively naphthenic fraction and fresh "make-up" solvent.

As aforementioned, the entrained matter, i. e., "dirty" solvent separated by centrifuge 36 is returned to chamber 11 through valve 39 or 40. In order to preserve equilibrium conditions in chamber 11 and to achieve maximum efficiency it is desirable that this dirty solvent be returned to chamber 11 at a point at which its composition, in terms of degree of extraction, is approximately the same as the composition of the solvent passing through chamber 11. In other words, this dirty solvent should be returned to a point at which it will augment the supply of the phase containing solvent and relatively naphthenic lubricating oil constituents without materially disturbing the equilibrium which exists at the point of introduction between solvent, relatively paraffinic constituents and relatively naphthenic constituents. This can be done by admitting the dirty solvent through valve 39 and/or valve 40, depending on which point corresponds more closely to the composition of the "dirty" solvent. Further alternative points of admission can, of course, be provided as required.

It will be understood that my process can readily be adapted to operate at any desired temperature by providing insulation, a steam jacket or a refrigeration jacket for casing 11 and/or by preheating or precooled one or both of the materials entering the process. Thus in solvent extraction work it is sometimes desirable to cool the solvent or the lubricating oil stock before admitting it to my process.

In the foregoing specific embodiment I have described the use of dichlorethylether in my process and apparatus as a desirable selective solvent. It will readily be comprehended, however, that a variety of other solvent materials can satisfactorily be used, the main requirement being a selective solvent power for the relatively paraffinic constituents or for the relatively naphthenic constituents of the original lubricating oil stock and a specific gravity substantially different from that of the lubricating oil stock to be treated. In general, it is the relatively naphthenic constituents which are selectively dissolved. As alternatives to the use of dichlorethylether, I find that nitrobenzene and phenol, among other substances, are very satisfactory in a solvent extraction process of this type. When operating on a wax-bearing lubricating oil stock I can either remove the wax before the solvent extraction or solvent extract the wax-bearing oil. In the latter cases it is sometimes necessary to expedite the flow of material through chamber 11, for instance, by means of screw conveyors in passages 23 and 27 as previously mentioned.

Throughout the foregoing description I have referred to the solvent as forming the lower stratum and the lubricating oil stock as forming the upper stratum. This is, of course, due merely to the relative specific gravities of these two materials and if it is desired to utilize a solvent having a lower specific gravity than that of the lubricating oil stock, my apparatus can readily be modified to permit this, the only essential difference being that the relative positions of the two strata are reversed.

Although in general the introduction of and removal of material from my apparatus should be such as to keep the interface between the two counter-current streams substantially in the center of chamber 11, it will be apparent that this can be adjusted to any other level if desired. Thus, for instance, if one of the two materials separates more readily from the central emulsified stratum than does the other, it is desirable to have at any given time in chamber 11 less of the material which separates more readily and more of the material which separates less readily, the result being that the separation of the latter is promoted. The relative amounts of the two materials in chamber 11 at any given time can readily be controlled by an adjustable float-controlled mechanism operating on one or more of the valves controlling the admission and/or removal of materials from my apparatus. Suitably this may consist of a device intermediate in density between the two materials in sump 12 which will float at the interface and control the operation of, say, valve 26 so as to keep the interfacial level at any desired point.

Although I have described my invention with particular reference to the solvent extraction of mineral lubricating oil stocks it will readily be understood that it may in whole or in part be applied to any process in which it is desired to contact two immiscible or partially miscible liquids, or other materials which can be flowed. Thus, for example, my process and apparatus can be used in the solvent extraction of vegetable oils, solvent extraction of motor fuels, the chemical treatment of various liquids with various reagents, the "washing" of liquids, etc. I do not, therefore, desire to be limited by my specific disclosure but only by the appended claims in which I have defined the novel features of my invention.

I claim:

1. A process for the counter-current contacting of two liquids comprising introducing one of said two liquids into an end contact zone forming one of a series of at least five adjacent linearly disposed contact zones, introducing the other of said two liquids into the end contact zone of said series of contact zones located opposite said first-mentioned end contact zone, flowing each of said two liquids through each of said contact zones in succession in a direction opposed to the direction of flow of the other of said two materials, separating said two liquids from each other at each of the borders between each adjacent pair of said contact zones, partially emulsifying all of the liquid passing through each of the intermediate contact zones, and separately withdrawing two resultant liquids from opposite end contact zones.

2. A process for the counter-current extraction of a mineral lubricating oil stock comprising relatively paraffinic constituents and relatively naphthenic constituents with a selective solvent for said relatively naphthenic constituents, comprising flowing said lubricating oil stock and said selective solvent in counter-current relationship to each other, washing the extracted lubricating oil stock by admitting at least a part of said selective solvent at a point near the point of withdrawal of said extracted lubricating oil stock, separately withdrawing the selective solvent after extraction of said relatively naphthenic constituents, separating said relatively naphthenic constituents from said selective solvent and returning said selective solvent to the process, centrifuging the resultant lubricating oil stock to remove entrained "dirty" solvent and returning said dirty solvent to the process at a point at which the degree of extraction is approximately that represented by the composition of the "dirty" solvent.

3. In apparatus for the countercurrent contacting of two liquids, a generally horizontal elongated chamber, a series of transverse baffles provided with openings at their tops and bottoms, said baffles partially dividing said chamber into two end compartments and a plurality of intermediate compartments, said passages which permit liquid strata at the top and bottom of said chamber to flow from one of said compartments to the next, a second series of transverse baffles, the members thereof located between the members of said first mentioned series of baffles and extending upward from the bottom of said chamber and downward from the top of said chamber, agitation means located within and confined to said intermediate compartments, means for introducing one of said two liquids into one of said end compartments, means for introducing the other of said two liquids into the other of said end compartments and means for withdrawing a resultant liquid from each of said end compartments.

4. In apparatus for the countercurrent contacting of two liquids a generally horizontal elongated chamber, a series of baffles, the members thereof extending downward from the top of said chamber at intervals along its length, passages located in the upper portions of the members of said series of baffles, a second series of baffles, the members thereof located in staggered relation to the members of said first-mentioned series of baffles at intervals along the length of said chamber and extending upward from the center of said chamber to points above the lowest points to which the

members of said first-mentioned series of baffles extend, but not extending to the top of said chamber, a third series of baffles, the members thereof extending upward from the bottom of said chamber at intervals along its length, passages located in the lower portions of the members of said third series of baffles, a fourth series of baffles, the members thereof located in staggered relation to the members of said third series of baffles at intervals along the length of said chamber and extending downward from the center of said chamber to points below the highest points to which the members of said third series of baffles extend, but not extending to the bottom of said chamber, agitation means located within at least some of the spaces between said various baffles and means for introducing said two liquids near opposite ends of said chamber and means for withdrawing a resultant liquid near each end of said chamber.

5. In apparatus for the counter-current contacting of two liquids, a generally horizontal elongated chamber, a series of transverse baffles provided with openings at their tops and bottoms, said baffles dividing said chamber into two end compartments and at least three intermediate compartments, said compartments communicating with each other through said passages which permit liquid strata at the top and bottom of said chamber to flow from each of said compartments to the next adjacent compartment, a series of baffles extending upward from the bottom of said chamber and downward from the top of said

chamber, the members of said last-mentioned series of baffles located between the members of said first-mentioned series of baffles, rotary agitation means located within and confined to said intermediate compartments, means for selectively adjusting the rate of rotation of said agitation means, means for introducing one of said two liquids into one of said end compartments, means for introducing the other of said two liquids into the other of said end compartments, and means for withdrawing a resultant liquid from each of said two end compartments.

6. A process for the counter-current extraction of an oil stock comprising relatively paraffinic constituents and relatively naphthenic constituents with a selective solvent for said relatively naphthenic constituents, comprising flowing said oil stock and said selective solvent in counter-current relationship to each other, washing the extracted oil stock by admitting at least a part of said selective solvent at a point near the point of withdrawal of said extracted oil stock, separately withdrawing the selective solvent after extraction of said relatively naphthenic constituents, separating said relatively naphthenic constituents from said selective solvent and returning said selective solvent to the process, separating the resultant oil stock from entrained "dirty" solvent and returning said "dirty" solvent to the process at a point at which the degree of extraction is approximately that represented by the composition of the "dirty" solvent.

ROBERT E. WILSON,

CERTIFICATE OF CORRECTION.

Patent No. 2,029,688.

February 4, 1936.

ROBERT E. WILSON.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, second column, line 48, claim 3, after the comma and before "said" insert the words said compartments communicating with each other through; and lines 68, 69 and 70, claim 4, strike out the words and comma "passages located in the upper portions of the members of said series of baffles,"; page 5, first column, lines 5, 6 and 7, claim 4, strike out the words and comma "passages located in the lower portions of the members of said third series of baffles,"; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 7th day of April, A. D. 1936.

Leslie Frazer

Acting Commissioner of Patents.

Seal)