Feb. 28, 1939. J. M. WHITELEY ET AL 2,148,716

TREATMENT OF HEAVY HYDROCARBON OILS WITH LIGHT HYDROCARBONS

Filed May 23, 1932 4 Sheets-Sheet 1

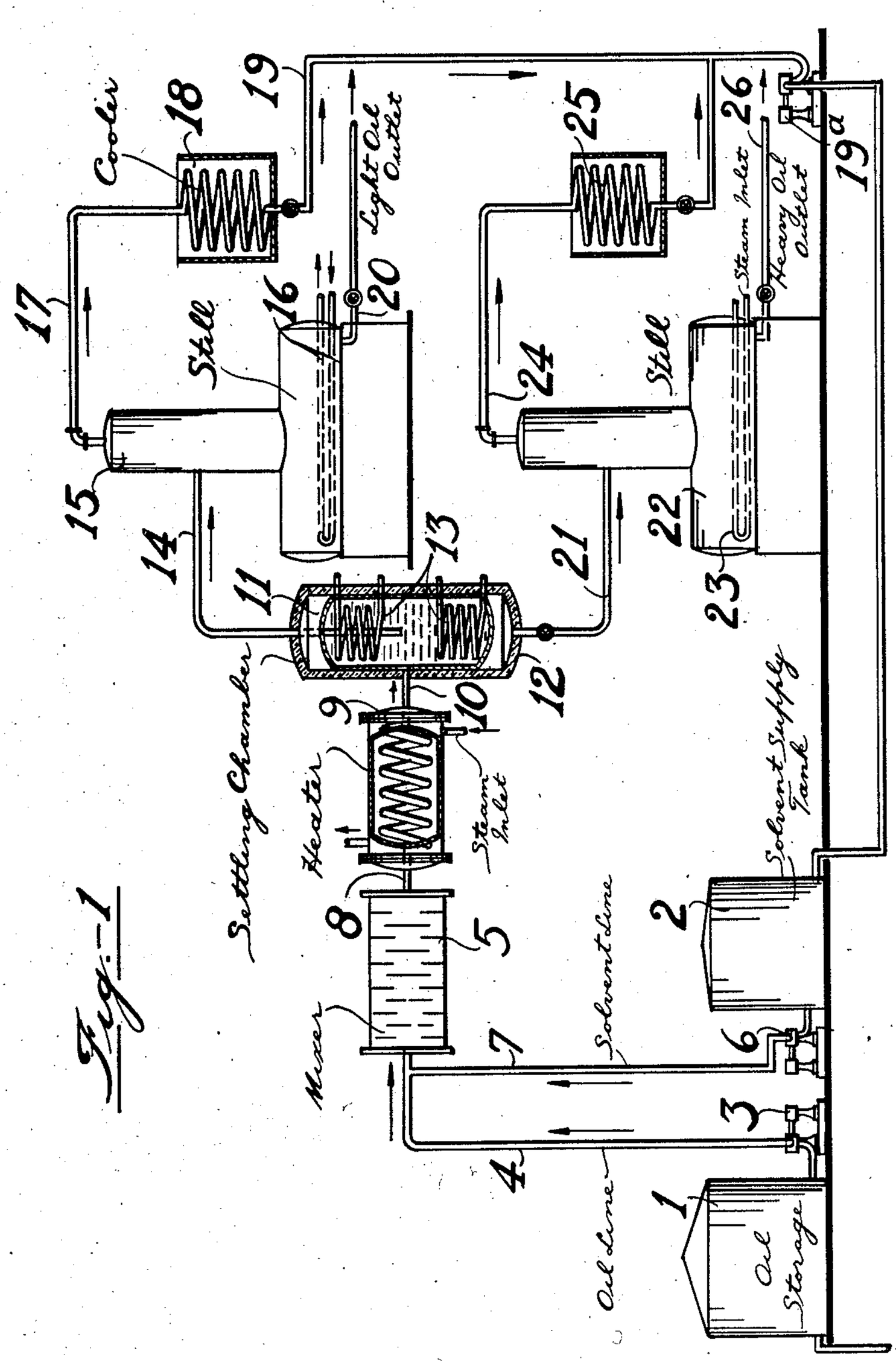


Fig. 1

James M. Whiteley INVENTORS
Gustav A. Beiswenger
BY
W. E. Currie ATTORNEYS.

Feb. 28, 1939. J. M. WHITELEY ET AL 2,148,716

TREATMENT OF HEAVY HYDROCARBON OILS WITH LIGHT HYDROCARBONS

Filed May 23, 1932 4 Sheets-Sheet 2

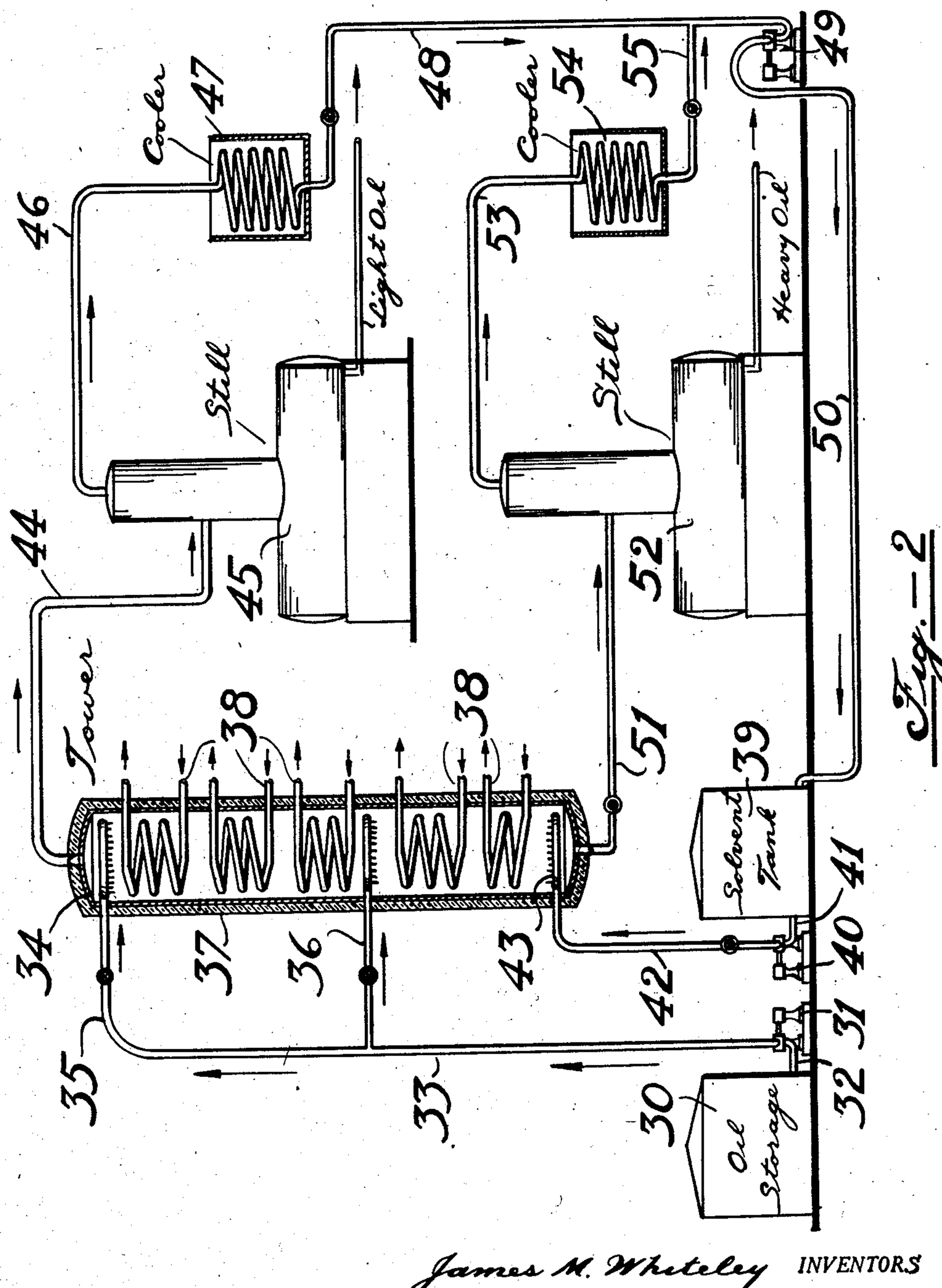


Fig. 2

James M. Whiteley INVENTORS
Gustav A. Beeswenger
BY
W. E. Currie ATTORNEYS.

Feb. 28, 1939. J. M. WHITELEY ET AL 2,148,716

TREATMENT OF HEAVY HYDROCARBON OILS WITH LIGHT HYDROCARBONS

Filed May 23, 1932 4 Sheets-Sheet 3

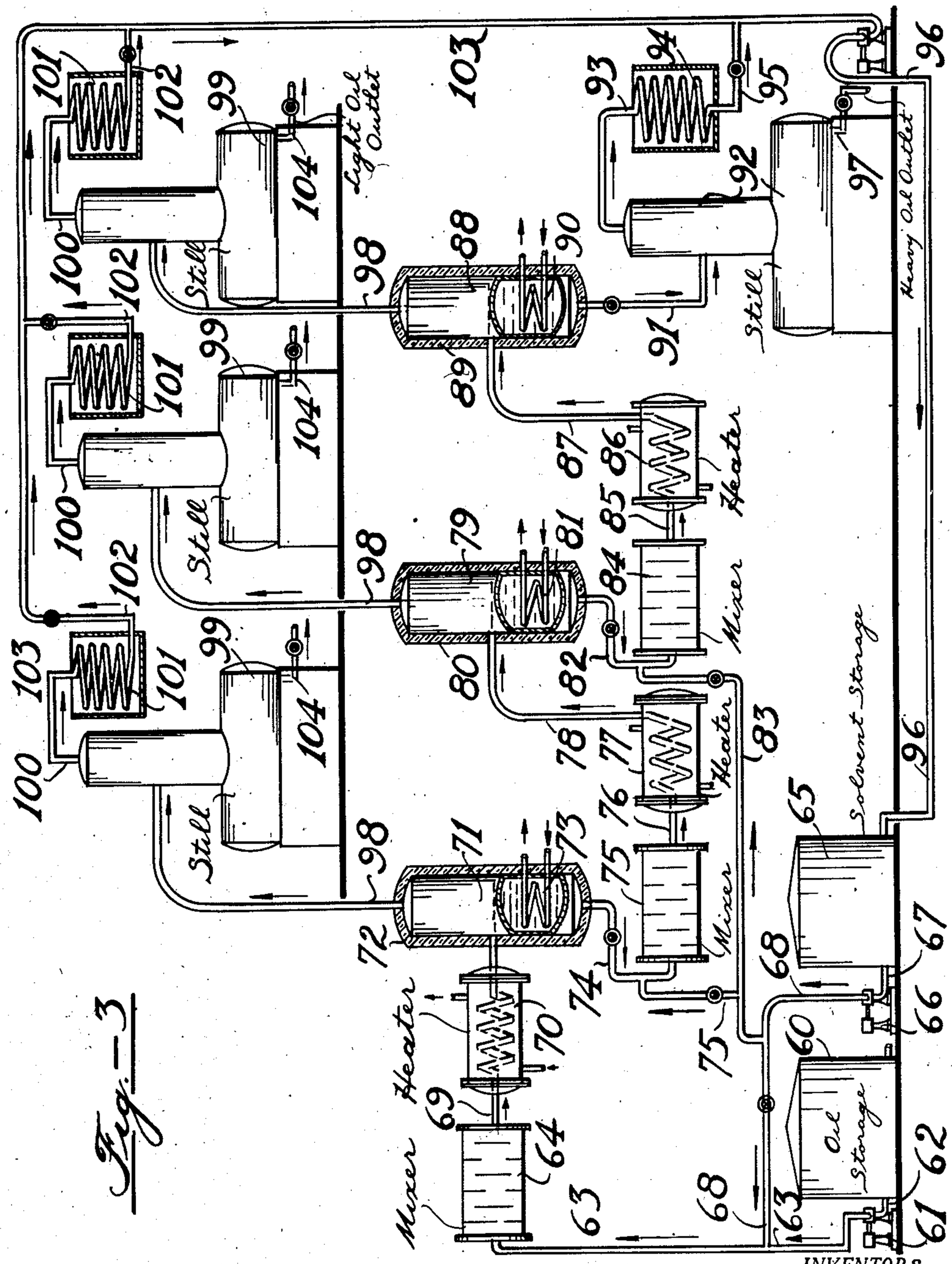


Fig. 3

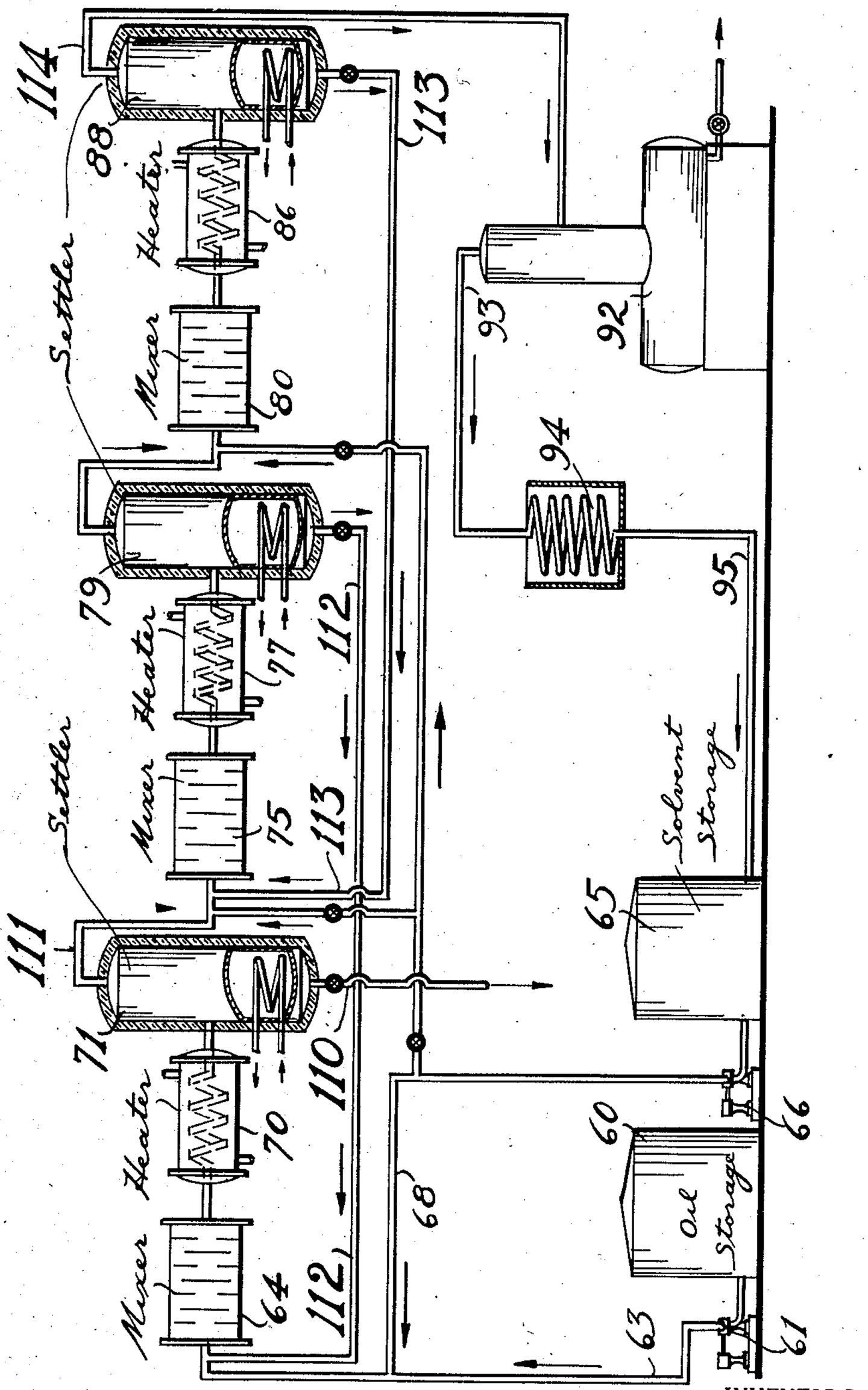
INVENTORS
James M. Whiteley
Gustav A. Beiswenger
BY
W. E. Currie ATTORNEYS.

Feb. 28, 1939. J. M. WHITELEY ET AL 2,148,716

TREATMENT OF HEAVY HYDROCARBON OILS WITH LIGHT HYDROCARBONS

Filed May 23, 1932 4 Sheets-Sheet 4

Fig. 4



INVENTORS
James M. Whiteley
Gustav A. Beiswenger
BY
W. E. Currie
ATTORNEYS.

Patented Feb. 28, 1939

2,148,716

UNITED STATES PATENT OFFICE

2,148,716

TREATMENT OF HEAVY HYDROCARBON OILS WITH LIGHT HYDROCARBONS

James M. Whiteley, Roselle, and Gustav A. Beiswenger, Elizabeth, N. J., assignors to Standard Oil Development Company, a corporation of Delaware

Application May 23, 1932, Serial No. 612,908

18 Claims. (Cl. 196—13)

This invention relates to hydrocarbon oils, especially petroleum oils, and more particularly relates to the treatment of these oils with light hydrocarbons.

The term light hydrocarbons will be understood to mean hydrocarbons or mixtures of hydrocarbons of 1 to 5 carbon atoms in a liquid or liquefied condition.

We have found that light hydrocarbons have, at high temperatures, say, within 50 to 100° F. of their critical temperature, a marked selective action on hydrocarbon oils. We have also found that the selective action of these light hydrocarbons may be varied by changing the temperature at which they are used.

In the present invention the light hydrocarbon solvent may comprise either a single light hydrocarbon or a mixture of light hydrocarbons and instead of changing the composition of the solvent to vary its selectivity (in accordance with the method described by James M. Whiteley in United States Ser. No. 582,501 filed December 21, 1931), the temperature at which it is used may be changed and the composition may remain substantially constant.

The present invention therefore comprises the treatment of hydrocarbon oils with light hydrocarbon solvents either once at a constant high temperature whereby the oil is separated into two components of dissimilar characteristics, or repeatedly at a progressively changing high temperature whereby each component may be separated further into other components.

The method of carrying out these treatments with the light hydrocarbons will be fully understood from the following description read with reference to the accompanying drawings of which

Figure 1 shows in sectional elevation a type of apparatus suitable for carrying out a single treatment at high temperature,

Figure 2 shows in sectional elevation a type of apparatus suited for a counter-current continuous treatment with a solvent of progressively changing temperature.

Figure 3 shows in sectional elevation a type of apparatus suited for an intermittent repeated extraction of the oil with a solvent of pregressively changing temperature, and

Figure 4 shows in sectional elevation a type of apparatus suited for a counter-current treatment in a series of stages with a solvent of progressively increasing temperature.

Referring to Figure 1, numeral 1 designates a storage supply tank for oil to be treated. Numeral 2 designates a supply tank for light hydrocarbon solvent. Pump 3 draws oil from tank 1 and forces it through line 4 into mixer 5, and pump 6 draws solvent from tank 2 and forces it through line 7 also into mixer 5, wherein oil and solvent are thoroughly commingled. From mixer 5, the thoroughly mixed oil and solvent flow through line 8 into a heating means 9 wherein the mixture is raised to within 50 to 100° F. of the critical temperature of the light hydrocarbon solvent. Heat may be provided by steam, by heat exchange, or by other means. From heating means 9 the hot mixture flows through line 10 into a settling chamber 11 provided with insulating means 12 and fitted with steam coils 13 whereby the required temperature can be maintained. The oil will separate into two layers in chamber 11, the top layer containing the lighter fractions of the oil and the bottom layer the heavier fractions. The top layer is withdrawn from chamber 11 through line 14 and is discharged into a still 15 provided with a steam coil 16 or other heating means whereby the light solvent may be distilled off of the oil. The vapors of solvent pass out of still 15 through line 17, are cooled in cooler 18 and the condensed solvent is returned to solvent supply tank 2 through line 19 by pump 19a.

The light oil freed from solvent is withdrawn from still 15 through line 20 and may be passed to storage or to equipment for further treating.

The bottom layer in separating chamber 11 is withdrawn therefrom through line 21 and passed into still 2 fitted with steam coil 23. The solvent vapors pass out of still 22 through line 24, pass through color 25 and the condensed solvent is returned to solvent supply tank 2 through line 19.

The heavy oil remaining in still 22 is withdrawn therefrom through line 26 and passed to storage or to equipment for further treating.

Referring to Figure 2, numeral 30 designates a storage supply tank for oil to be treated. Pump 31 draws oil from tank 30 through line 32 and forces it through line 33 either into the top of tower 34 by means of line 35 or into the middle of tower 34 by line 36. If the oil is discharged into the tower at the top the lighter fraction of the oil which separates out leaves the tower immediately and the counter-current extraction is then carried out only on the heavier fraction of the oil; but if the oil is introduced into the middle of the tower the extraction will be carried out on both the light and the heavy fractions of the oil, the former with a solvent of progressively decreasing density and the latter with a solvent of

progressively increasing density all as will be more fully explained below.

Tower 34 is an elongated vertical chamber which provides for counter-current flow of oil and solvent. It may be packed with contacting means such as chain, jackstones, lumps of clay, or suitably designed plates, or may be without such contacting means. It may be provided with an insulating coating 37, and with heating means such as steam coils 38 placed at intervals along its length by means of which the temperature in the various sections of the tower may be independently controlled.

Numeral 39 designates a storage supply tank for the light hydrocarbon solvent. Pump 40 draws solvent from tank 39 through line 41 and forces it through line 42 into the base of tower 34 into which it may be discharged through a suitable series of openings indicated at 43.

In the tower the oil flows downwardly in counter-current relation to the upwardly rising stream of solvent the temperature of which is progressively increased as it rises by means of steam coils 38. The oil is thereby separated into a lighter soluble fraction and a heavier insoluble fraction. The former flows out of tower 34 through line 44 and discharges into a still 45 wherein the solvent is separated from the oil by distillation. Vapors of solvent pass out of still 45 through line 46, flow through cooler 47 wherein they are condensed, and the recovered solvent is returned by line 48, pump 49 and line 50 to solvent storage tank 39.

The heavier fraction of the oil passes out of tower 34 through line 51 and discharges into still 52 wherein the solvent is removed by distillation. Vapors of solvent pass out of still 52 through line 53, flow through cooler 54, and the condensed solvent returned to solvent supply tank 39 by lines 55 and 48, pump 49 and line 50.

Referring to Figure 3, numeral 60 designates a storage supply tank for oil to be treated. Pump 61 draws oil from tank 60 through line 62 and forces it through line 63 into a mixing device 64. Numeral 65 designates a solvent supply tank. Pump 66 withdraws solvent from tank 65 through line 67 and forces it through line 68 into mixing device 64 wherein oil and solvent are intimately commingled.

The mixture of oil and solvent then flows through line 69 into a heating means 70 wherein the mixture is raised to a temperature within say 25° F. or less of the critical point of the solvent. The heat may be supplied by steam or other hot gases. The heated mixture discharges into a settling chamber 71 fitted with an insulating coating 72 and a heating coil 73 wherein the mass is allowed to stand. The oil separates into two layers, the top layer containing the lighter fractions and the bottom layer the heavier.

The bottom layer is withdrawn from settling chamber 71 through line 74. It passes to a second mixing device 75 wherein it may be commingled with additional solvent supplied thereto through line 75. The mixed oil and solvent then flow through line 76 to a heating means 77 wherein the mixture is heated, this time to a temperature somewhat lower, say 10 to 15° F. lower, than it was heated prior to entering the first settling chamber. The heated mixture flows through line 78 and discharges into a second settling chamber 79 provided like the first one with an insulating coating 80 and a heating coil 81. After standing until the two layers separate, the bottom layer is withdrawn through line 82 and subjected again to another extraction. This time with a solvent of still lower temperature. Additional solvent is supplied through line 83 and the oil and solvent pass through mixing device 84, line 85, heating means 86 and line 87 into a third settling chamber 88, provided with insulating coating 89 and heating coil 90. A third separation occurs and the bottom layer may be withdrawn and subjected to as many further extractions with solvent at progressively lower temperatures as desirable.

If settling chamber 88 is taken as the last chamber, the bottom layer will be withdrawn therefrom through line 91 and discharged into a still 92 wherein the solvent may be separated from the oil by distillation. Solvent vapors pass out of the still through line 93, flow through cooler 94, and the condensed solvent returns to solvent supply tank 65 through lines 95 and 96. The heavy oil remaining in the still is withdrawn therefrom through line 97.

The top layers are withdrawn from the several settling chambers through lines 98 and discharge into stills 99 wherein the solvent is separated from the oil by distillation. Solvent vapors pass out of stills 99 through lines 100, and flow through coolers 101. The condensed solvent then returns to solvent supply tank 65 through lines 102, 103 and 96. The light oil remaining in stills 99 is withdrawn therefrom through lines 104. The several fractions of light oil so obtained may be blended in any desired proportions, or may be maintained separate and worked up into several different light oil fractions. It will be understood that in place of using a separate still for the top layer from each settling chamber, a single still into which the top layers from all the settling chambers discharge may be used. Any other arrangement of stills may be made.

Referring to Figure 4 which illustrates a type of apparatus for carrying out a counter-current treatment of both the lighter and heavier fractions in a series of stages, the apparatus is in general similar to that shown in Figure 3 with these exceptions: The bottom layer formed in the first settler instead of being forwarded to the second mixer and settler is removed through line 110; the top layer formed in the first settler is withdrawn through line 111 and passed to the second mixer and settler; the bottom layers from the second and third settlers are withdrawn through lines 112 and 113 respectively, and introduced into the mixers ahead of the first and second settlers respectively. In other words, the bottom layers are continuously withdrawn and returned to the next preceding stage instead of being forwarded to the next succeeding stage as in Figure 3, and the top layer in each settler is forwarded to the next succeeding stage instead of being removed as in Figure 3.

The top layer from the final settler is removed through line 114 and passed into a still wherein the oil is separated from the solvent. In this method of operation the final top layer comprises an oil of greatly improved color over the original oil. It should be noted that in this method of operation the temperature is progressively increased from stage to stage instead of progressively decreased as is the case in treating the bottom layer in successive stages as in Figure 3. In other respects the apparatus in Figure 4 may be substantially similar to that in Figure 3, allowing for obvious modifications.

In the operation of our process the principal variable factors are the type of material selected

as the feed oil, the kind of light hydrocarbon solvent, the proportion of solvent to oil, the temperature at which the treatment is carried out and the pressure maintained.

In general, our process is applicable to any type of heavy hydrocarbon material, whether obtained from petroleum oil or its products of distillation or cracking, or from the products of the destructive distillation or hydrogenation of petroleum oils, coals, tars, pitches, shales, lignites, bitumens and the like. Our process is also applicable to synthetic hydrocarbon oils, waxes or resins, prepared for example by condensation or polymerization processes. It will be understood that at the temperatures at which the extraction is carried out, most of the normally solid hydrocarbons are above their melting points and therefore have substantially the same solubility characteristics as oils. Our process is particularly adapted, however, to the fractionation, purification and decolorization of petroleum oils, especially the lubricating fractions thereof.

The type of light hydrocarbons that may be used as the solvent in our process comprise generally hydrocarbons of 1 to 5 carbon atoms or mixtures of any 2 or more of such hydrocarbons. Thus methane, ethane, propane, butane, pentane, ethylene, propylene, butylene, amylene, and isomers of these may be used. The presence of small quantities of higher molecular weight hydrocarbons is not especially harmful but in general it is desirable to avoid the presence of these higher hydrocarbons. Ethane, propane and butane, or mixtures of ethane and propane or propane and butane are particularly satisfactory solvents for our purposes. The gases obtained in the cracking of petroleum distillates and in the stabilization of gasolines are generally rich in the lighter hydrocarbons such as ethane, propane and butane and they furnish a convenient and readily available source of the solvent hydrocarbons.

The proportion of solvent to all may be varied within wide limits, but between 3 and 15 volumes of solvent per volume of oil is satisfactory for most purposes. Between 8 and 12 volumes of solvent per volume of oil is an especially suitable proportion.

The temperatures employed in the operation of the process in general range from the critical temperature to 10, 50, 100, 125° F. or more below the critical temperature of the particular light hydrocarbon selected as the solvent. Thus for propane which has a critical temperature of about 212° F., temperature may be decreased progressively from say 200° F. to 175 to 150 to 100 to 75° F., and so on, the density and consequently the selectivity thereof increasing and decreasing respectively with the decreasing temperature. For butane and pentane the temperatures of operation will be correspondingly higher and may be readily determined from the critical temperature of each.

When using solvents comprising mixtures of two or more light hydrocarbons, temperatures higher than those used with the lighter of the two or more hydrocarbons alone are generally required in order to obtain the same selectivity. Thus when using a mixture of propane and butane, the temperature necessary to obtain the same selectivity as exhibited by propane alone at a particular temperature must be near or above the critical temperature of propane but below the critical temperature of butane. For example, a heavy bottoms oil obtained from a Ranger crude is treated with 8 volumes of propane per volume of oil at a temperature of about 158° F. and an 80% yield of an oil having a Saybolt viscosity at 210° F. of 114 seconds and a color (Robinson) of 2½ (dilute) is obtained. When a solvent comprising 8 parts of propane to 1 part of butane is used it is necessary to treat at a temperature of 180° F. in order to obtain the same yield of the same quality oil.

Similarly when using a solvent comprising ethane and propane, higher temperatures are necessary than if ethane alone is used in order to obtain the same selectivity in each case.

The pressure in our process should in general be maintained sufficiently high to retain the light hydrocarbons in liquid phase at the temperature of working, but preferably not substantially greater than the equilibrium vapor pressure of the liquid at that temperature. This pressure will be between say slightly above atmospheric and 50 or more atmospheres depending upon the particular components of the solvent and the temperature at which the treatment is carried out. If the solvent comprises a mixture of light hydrocarbons, the pressure necessary will be close to the equilibrium vapor pressure of the lightest component of the solvent which is present in substantial amount.

The type of operation indicated in Figure 1, that is, a single extraction with solvent at a high temperature is especially adapted for obtaining from a heavy oil a fraction of better quality with respect to viscosity temperature characteristics and gravity. It is also adapted for separating a heavy lubricating fraction into a lighter fraction and a heavier fraction, both of which fractions differ in characteristics from the original oil.

The types of operation indicated by Figures 2, 3 and 4, that is, counter-current or repeated intermittent extraction, are especially adapted for decolorizing and highly purifying heavy petroleum oil fractions and obtaining therefrom valuable lubricating oils. By means of the successive treatment with solvent of progressively changing density it is possible to extract from the oil solid impurities which could not be removed in a single extraction. In the case of intermittent extraction it is possible to obtain a plurality of oil fractions of dissimilar characteristics, and these may be blended in any proportions to obtain blended oils of any desired characteristics. It is also possible by intermittent extraction to obtain in the last steps oils of extremely high viscosities, say from 1000 to 5000 seconds Saybolt viscosity at 210° F. Oils of these high viscosities cannot ordinarily be obtained by the usual fractionating means due to the fact that they decompose or break down at the temperatures necessary to vaporize them even when distilled under high vacuum. Moreover, all of the several fractions into which the heavy oil is separated by our process are characterized by much greater stability to heat and oxidation than fractions obtained by distillation.

Prior to treatment according to this process, the oils may be subjected to preliminary purification treatments. Thus oils initially rich in asphaltic bodies may first be treated to remove a substantial portion of these materials. One method of removing asphaltic bodies which is especially convenient in connection with the present process is to treat the oil with liquefied hydrocarbons such as propane or propane and ethane at normal temperatures, say around 50 to 100° F. In this way the asphaltic bodies are thrown out

of the oil in a hard, granular substantially oil-free condition and the remaining oil is already in solution in the light hydrocarbon used in the high temperature treatment of the process herein described. Other methods of removing asphalt may of course be used.

The oils may also be subjected to hydrogenation, phenol extraction, aluminum chloride digestion, acid and clay treatment and so forth either preceding or following subjection to the light hydrocarbon solvent treatment. Dewaxing also may precede or follow the solvent treating, and any suitable method of dewaxing may be used.

Following the purification of the material in accordance with the method outlined above, and before removing the solvent therefrom, a still further purification may be conveniently and advantageously effected by filtering the flux while still at the high treating temperature through a bed of adsorptive material such as clay, charcoal and the like. Filtration of the oil or other hydrocarbon material while in hot light hydrocarbon solution proceeds at an extremely rapid rate and substantially larger yields of oil per ton of clay are obtained than can be obtained by filtration in naphtha solution. The extent of the additional purification moreover is substantial, and the oils filtered in this manner are comparable in purity and color to oils obtained only after repeated subjection to other methods of purification.

It will be understood of course that the hydrocarbon material may be filtered through the solid adsorptive media while in hot light hydrocarbon solution without a preliminary purification such as described above. This would be particularly advantageous if the initial material contains only a relatively small amount of colored or normally solid bodies.

As an illustration of the type of operation shown in Figure 1, a heavy dark-colored residuum obtained from a Ranger crude is first partially purified by treatment with propane at 80° F. By this treatment a large proportion of the asphaltic material is precipitated from the residuum. The partially purified oil then has the following characteristics:

Gravity, A. P. I.	degree	22.5
Saybolt viscosity @ 210° F.	seconds	133.4
Conradson carbon		2.48
Color, Robinson, ¾ (dilute).		

This oil is then diluted with 8 volumes of propane and the mixture heated to 183° F. whereupon two layers form. After allowing the mixture to stand at this same temperature the two layers are separated and the propane distilled off from each. The two oil fractions so obtained have the following characteristics:

	Top layer	Bottom layer
Gravity, A. P. I.	24.6°	20.1°
Saybolt viscosity @ 210° F.	105.5 seconds	229 seconds
Conradson carbon	1.98	5.67
Color, Robinson	3½ (dilute)	Very dark
Yield	68%	32%

The following table illustrates the effect of varying the temperature on the selectivity of the light hydrocarbon. The oil used is the same Ranger residuum as was used in the above example. The light hydrocarbon comprises propane and is used in the proportion of 8 volumes of propane to 1 volume of oil. The pressures maintained at each temperature are substantially the saturated vapor pressures of propane at those temperatures:

Temp. °F.	Pressure lbs./sq. in.	Yield of oil	Saybolt viscosity @ 210° F.	Color (Robinson) (dilute)	Weight percent oil in top layer
		Percent	*Seconds*		
80	160	85	135	1	21.5
131	340	88	130	1¼	21.3
149	375	81.4	114	2¼	19.7
162	400	75	100	2½	17.9
185	500	56	86	5¼	14.2

This invention is not limited by any theories of its mechanism nor by any details or data which have been given merely for purposes of illustration, but is limited only in and by the following claims in which we wish to claim all novelty inherent in the invention.

We claim:

1. Process of treating heavy hydrocarbon oil which comprises diluting the oil with a light normally gaseous hydrocarbon solvent maintaining said normally gaseous hydrocarbon solvent in liquid state whereby said oil is caused to separate into a dissolved layer and an undissolved layer removing the undissolved layer, heating the remaining dissolved layer to a temperature near the critical temperature of the light hydrocarbon solvent, whereby the dissolved layer is further separated into two oil layers, separating the two layers and repeatedly subjecting the bottom layer to the same treatment at progressively lower temperature, the final temperature being substantially above the wax separation temperature of the oil.

2. Process of treating heavy hydrocarbon oil which comprises diluting the same with a light hydrocarbon solvent comprising hydrocarbons of 1 to 5 carbon atoms, heating the mixture to a temperature near the critical temperature of the light hydrocarbon solvent, allowing the mixture to separate into two layers, removing the bottom layer, adding more light hydrocarbon solvent thereto, heating this mixture to a temperature somewhat lower than that to which the original mixture was heated, allowing this mixture to separate into two layers, removing the bottom layer, adding more hydrocarbon solvent thereto, heating to a temperature lower than that to which the second above mixture was heated, allowing this mixture to separate into two layers, removing the bottom layer and recovering the heavy oil therefrom.

3. Method according to claim 1 in which the initial temperature of the solvent is within 10° F. of the critical temperature of the solvent and the final temperature is within 100° F. of the critical temperature of the solvent.

4. Method according to claim 1 in which pressure is maintained sufficient to retain the light hydrocarbon solvent in liquid phase but not substantially greater than the equilibrium vapor pressure at the temperature of working.

5. The method of obtaining valuable lubricating oils from a heavy petroleum oil fraction which comprises subjecting the heavy oil to repeated extraction with a light hydrocarbon solvent comprising propane, the temperature of which is progressively decreased from near the critical temperature of propane to about 100° F. below that temperature, and recovering the oil from the several extracts so obtained.

6. The method of fractionating a heavy oil by

means of a light hydrocarbon consisting principally of propane, which method comprises intimately admixing said oil with about 6 to 8 volumes of the light hydrocarbon solvent, introducing said mixture into a settling zone at a temperature from about 130° F. to 185° F. and under a pressure of about 350 pounds to 500 pounds per square inch, separating an upper light oil layer from a lower heavy oil layer, removing one layer from the other, and removing the light hydrocarbon from each layer.

7. The method of claim 6 wherein the heavy oil layer is admixed with further amounts of light hydrocarbon and subjected to a further high temperature separation for the removal of further amounts of light oil therefrom.

8. The method of separating a heavy hydrocarbon oil into normally liquid fractions of different characteristics which comprises diluting the oil with a liquefied normally gaseous hydrocarbon solvent, heating the mixture to a temperature between the critical temperature of the solvent and about 50° F. below the critical temperature at which the oil is caused to separate into a soluble liquid oil fraction and an insoluble liquid oil fraction, separating the two layers and recovering the oil therefrom.

9. The method according to claim 8 in which the liquefied normally gaseous hydrocarbon solvent contains a substantial proportion of hydrocarbons having two and three carbon atoms.

10. The method according to claim 8 in which sufficient pressure is maintained to retain the liquefied hydrocarbon in the liquid phase.

11. The method of treating heavy hydrocarbon oil which comprises diluting the oil with several volumes of a liquefied normally gaseous hydrocarbon solvent, heating the mixture to a temperature between the critical temperature of the liquefied hydrocarbon solvent and about 50° F. below the critical temperature at which the oil is caused to separate into two liquid layers, removing the top layer, filtering the same through a bed of solid adsorptive media while maintained at substantially the same temperature as that at which the separation occurred, and recovering the oil from the filtrate.

12. The method of preparing a highly purified dewaxed lubricating oil from a heavy hydrocarbon oil containing asphalt and wax, which comprises diluting the oil with several volumes of a liquefied normally gaseous hydrocarbon, removing the asphaltic material thereby caused to precipitate, chilling the solution to a wax separation temperature, removing the wax so caused to separate, heating the remaining solution to a temperature between the critical temperature of the liquefied hydrocarbon and about 50° F. below the critical temperature at which the oil is caused to separate into a soluble liquid oil fraction and an insoluble liquid oil fraction, separating the two fractions, filtering the soluble fraction while still hot and dissolved in the liquefied hydrocarbon through a bed of clay, and recovering the purified oil from the filtrate.

13. The method of separating an asphalt-free hydrocarbon oil into normally liquid fractions of different characteristics which comprises diluting the oil with several volumes of a liquefied normally gaseous hydrocarbon solvent, heating the mixture to a temperature between the critical temperature of the solvent and about 50° F. below the critical temperature at which the oil is caused to separate into a soluble liquid oil fraction and an insoluble liquid oil fraction, separating the two layers and recovering the oil therefrom.

14. The method of separating a heavy hydrocarbon oil into a plurality of fractions of different characteristics by treatment with a liquefied normally gaseous hydrocarbon solvent in a plurality of stages which includes the steps of dissolving the oil in several volumes of the liquefied hydrocarbon, maintaining sufficient pressure to retain the liquefied hydrocarbon in the liquid phase, heating the solution to a temperature at which the solution is caused to separate into two distinct layers, separating the two layers, repeating the sequence of steps on the bottom layer in a plurality of stages, and recovering the oil from the several layers; the temperature in the successive stages being progressively decreased from a temperature in the first stage close to the critical temperature of the liquefied hydrocarbon solvent to a temperature in the final stage about 50° F. below the critical temperature of the liquefied hydrocarbon solvent.

15. The method according to claim 14 in which the liquefied hydrocarbon solvent comprises liquefied propane.

16. The method according to claim 14 in which the oil is dissolved in from 8 to 12 volumes of liquefied hydrocarbon.

17. The method of separating a heavy hydrobon oil into a plurality of oil fractions having different characteristics by treatment with a liquefied normally gaseous hydrocarbon solvent in a plurality of successive stages which includes the steps of dissolving the oil in several volumes of a liquefied normally gaseous hydrocarbon, heating the mixture to a temperature close to but below the critical temperature of the liquefied hydrocarbon at which temperature a portion of the oil becomes insoluble in the liquefied hydrocarbon, removing the insoluble portion, admixing it with additional liquefied hydrocarbon, heating it to a temperature lower than that at which the first separation occurred but within about 50° F. of the critical temperature of the liquefied hydrocarbon at which temperature another portion of the oil becomes insoluble in the liquefied hydrocarbon, removing the insoluble portion, admixing it with additional liquefied hydrocarbon, heating it to a temperature lower than that at which the second separation occurred, but within about 50° F. of the critical temperature of the liquefied hydrocarbon at which temperature a third separation of insoluble oil fractions occurs, separating the soluble fractions from the insoluble and finally recovering the oil from the several separated fractions.

18. The method of separating a heavy hydrocarbon oil into normally liquid fractions of different characteristics which comprises diluting the oil with a liquefied normally gaseous hydrocarbon solvent containing a substantial proportion of a hydrocarbon having three carbon atoms, heating the mixture to a temperature between the critical temperature of the solvent and about 50° F. below the critical temperature at which the oil is caused to separate into a soluble liquid oil fraction and an insoluble liquid oil fraction, separating the two layers and recovering the oil therefrom.

JAMES M. WHITELEY.
GUSTAV A. BEISWENGER.