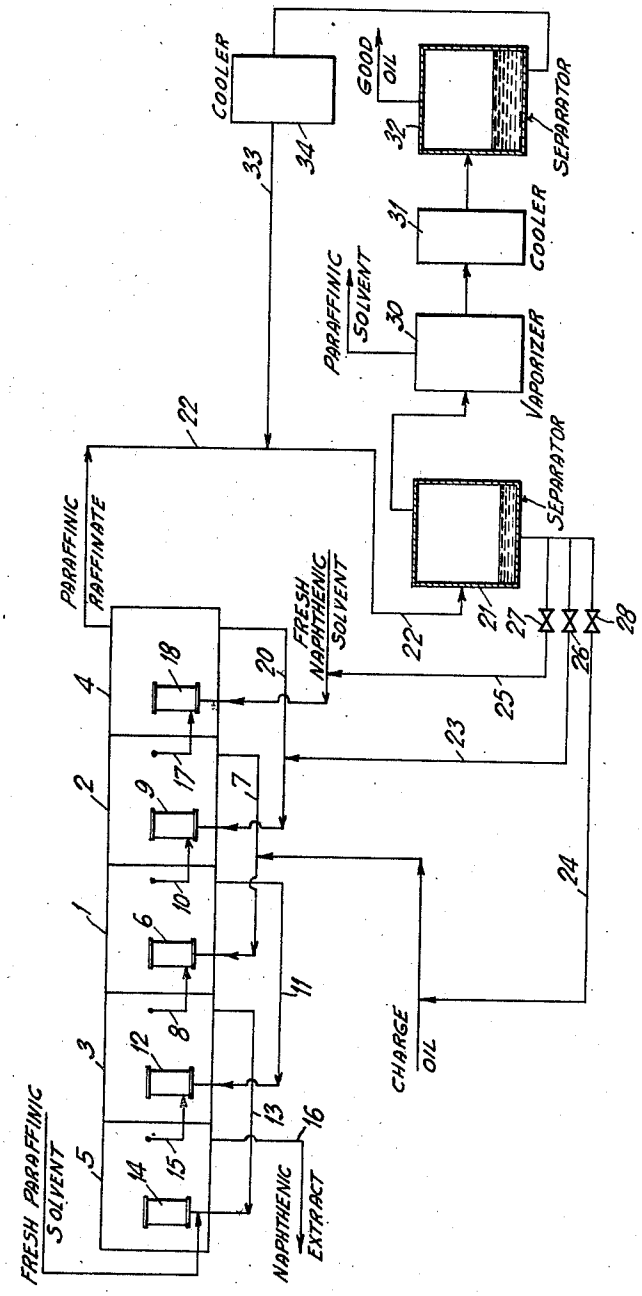
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PURIFICATION OF OIL FRACTION AND SOLVENT RECOVERY

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PURIFICATION OF OIL FRACTION AND SOLVENT RECOVERY

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8 Claims. (Cl. 196—13)

This invention relates to the refining or extracting of mineral oils by means of solvents. More specifically, the invention has to do with a process in which the charge oil to be refined is treated jointly with a solvent for paraffinic type oil and a solvent for naphthenic type oil and is thus caused to separate into layers or fractions respectively more praffinic and more naphthenic in character than the charge oil.

The solvents employed are advantageously, as the paraffinic solvent, a liquefied normally gaseous hydrocarbon such as propane (possibly containing ethane and butane), or normally liquid petroleum ether (largely hexane), and a naphthenic solvent such as cresylic acid or wood tar acid (for example guaiacol, creosol or beechwood creosote) as covered in my Patents 1,942,348 and 1,942,349, granted May 30, 1933. A particularly efficient naphthenic solvent, as described and claimed in my copending application Ser. No. 28,673, filed June 27, 1935, may comprise about 18–38% phenol, about 5% of the higher boiling homologues of pyridine, boiling from about 365°–405° F., as found in coal tars, and the balance cresols (ortho, meta and para) in usual relative proportions. About two parts by weight of each solvent per part by weight of oil are suitable proportions, for example. However, while the present invention is particularly advantageous in connection with the use of such solvents, it may also be useful in connection with other paraffinic and naphthenic solvents, for instance petroleum ether and nitrobenzene.

In my copending application Ser. No. 709,098, filed January 31, 1934, there is disclosed an improved process for separating from a paraffinic raffinate or upper layer fraction, produced by the combined action of such solvents, a large proportion of the naphthenic solvent which is entrained or otherwise carried along with the paraffinic raffinate leaving the main extraction system. It is highly desirable to reuse this separated naphthenic solvent in the main extraction, but it has been found that it contains a substantial amount of oil which is, in many if not all cases, still quite paraffinic as compared to the charge oil, although generally less paraffinic in character than the good oil produced as a result of the separation of naphthenic solvent from the raffinate.

When such a mixture of naphthenic solvent and oil is introduced directly into the extraction system at one end, where the mixture in the system is largely paraffinic solvent and paraffinic type oil, certain rather undesirable results may be produced, especially in connection with some types of charge oil. For instance, the settling rates at which the mixture separates into layers may be adversely affected. For another thing, some of the oil which is in solution in the naphthenic solvent may be thrown out of solution in this solvent on account of the presence of paraffinic solvent in the extraction chamber. Where the extraction process is conducted as a countercurrent operation, and especially where the solvents are introduced at opposite ends of the extraction system and counterflowed, the oil being introduced at an intermediate point, the tendency is for the reintroduced oil-naphthenic solvent mixture to unsettle the desired equilibrium at the end of the system, and when more and more of this mixture is thus reintroduced the disturbance of the desired equilibrium may spread through the entire system, slowing down the extraction or adversely affecting the yield and quality of the products, or both.

A feature of the present invention therefore resides in the provision of superior apparatus and process which avoid these disadvantages and result in a greater efficiency.

Other features, objects and advantages will become apparent or be pointed out in connection with the following detailed description of an example of the invention, reference being had to the accompanying drawing or flow sheet which illustrates diagrammatically a form of apparatus in accordance with the invention.

Referring to the drawing, the primary extraction system may preferably comprise a series of pots or chambers 1, 2, 3, 4 and 5, five being shown although any suitable number may be employed. Furthermore, other convenient extraction apparatus may be substituted, if desired, within the broader aspects of the present invention. When a series of chambers is employed the charge oil, which may be a crude mineral oil, distillate, residual topped stock, or other fraction, previously otherwise treated or not, and this charge oil may advantageously be introduced into pot 1 after being commingled within mixer 6 with a lower layer fed from pot 2 through line 7 and with an upper layer fed from pot 3 through line 8. Where the paraffinic solvent is a liquefied normally gaseous hydrocarbon the mixtures in the several pots preferably fill the same and the system is maintained under sufficient pressure to prevent vaporization of this solvent.

It is desirable to feed the lower layers from chamber to chamber and to introduce the charge oil and solvents to the system by means of suit-

able pumps (not shown), while the upper layers may be fed forward from chamber to chamber through nozzles which produce a jet action when the pressure in each chamber into which an upper layer is fed is lower than the pressure in the chamber from which it is fed, as is preferably the case. The flow of the upper layers may be also caused at least in part by the pumping of the lower layers into chambers of relatively high pressure. The pressure differentials may be small, for instance in the order of a few pounds per square inch.

The mixing chamber 6 may have suitable connections (not shown) to enable the mixture to flow into chamber 1, where said mixture is allowed to separate into layers by reason of the presence of solvents in the upper and lower layers introduced with the charge oil. The upper layer from chamber 1 may be passed into a mixer 9 connected with chamber 2, through line 10, while the lower layer from chamber 1 may be pumped into mixer 12 which is connected with chamber 3 by a line 11. The lower layer from chamber 3 may be pumped to chamber 5 through line 13 and mixer 14, where it meets fresh paraffinic solvent supplied from any convenient storage tank. In this end chamber 5 the paraffinic solvent frees the lower layer from chamber 3 (which layer is already quite naphthenic in character) of its remaining paraffinic type oil, and the upper layer thus formed may be passed through line into mixer 12, while the naphthenic extract or lower layer from chamber 5 is withdrawn through pipe and treated for recovery of solvents by evaporation or otherwise.

Similarly, the upper layer from chamber 2 passes through line 17 into mixer 18 connected with chamber 4, and meets fresh naphthenic solvent which extracts certain naphthenic constituents and forms a lower layer which is passed through line 20 to mixer 9. The paraffinic raffinate or upper layer produced in chamber 4 is drawn off. This raffinate contains much paraffinic type oil and paraffinic solvent, and also contains considerable naphthenic solvent.

At this point it may be remarked that any suitable values or other known contrivances may be applied to the apparatus thus far described for controlling the rates of flow, and so forth, but since such details may vary considerably and form no part of the present invention in its broader aspects, they are not described in detail.

The paraffinic raffinate from chamber 4, when propane and cresylic are employed, for example, is preferably at a temperature of about 70° F. to 90° F. depending somewhat upon other conditions. Such temperatures may be produced by employing hot solvents in the process. For instance, when one or both of the solvents is or are recovered by evaporation from the raffinates and extracts produced in the process, they absorb some quantities of heat and when they are returned to the extraction system they transfer some heat to the oil so that the mixtures in the chambers are thus heated. With solvents like propane and cresylic acid, such a temperature is advantageous since it is then desirable to conduct operations at a temperature not too far below the critical solution temperature (i. e., the temperature below which a separation into layers occurs in a mixture of oil and solvents) in order to promote the efficiency of extraction and to use minimum amounts of solvents consistent with desired results.

The paraffinic raffinate from chamber 4 is then preferably passed through line 22 into a separator 21 in mixture with naphthenic solvent produced as hereinafter described, whereupon, due to the presence of this additional naphthenic solvent, it separates into layers, the upper of which is primarily paraffinic oil and paraffinic solvent and the lower of which is primarily naphthenic solvent and some oil.

The lower layer from separator 21 is preferably returned to the primary extraction system between the point of introducing the charge oil and the point where the fresh naphthenic solvent is introduced, or advantageously at a point where the composition of the mixture in the system is substantially the same as the composition of the lower layer from separator 21. For instance, it may be fed through lines 23 and 20 to chamber 2. However, in the present system, in conjunction with certain features about to be described, the lower layer fraction in separator 21 is small and may be returned through line 24, along with charge oil, to chamber 1, or even through line 25, along with fresh naphthenic solvent, to chamber 4. Suitable valves 26, 27 and 28 may be provided for controlling at will the return of this lower layer.

The upper layer from separator 21 is next preferably passed to a suitable evaporator 30, where the paraffinic solvent, which vaporizes at a markedly lower temperature than the naphthenic solvent, is removed and recovered. The remaining oil thus produced still contains considerable naphthenic solvent and is sent to cooler where its temperature is reduced sufficiently to cause it to separate into layers in separator 32, the good oil forming an upper layer, while the lower layer consists mainly of naphthenic solvent. Any relatively small amount of naphthenic solvent remaining in the good oil may be readily distilled or otherwise removed therefrom. This lower layer may be passed via line 33 to the paraffinic raffinate and when this mixture is introduced into separator 21, as hereinabove described, it separates into layers. If desired, the material in line 32 may be cooled by a cooler 34 to about the temperature of the raffinate.

Separator 21 is thus, in a sense, an extraction chamber where the lower layer from separator 32 (which layer is relatively rich in naphthenic solvent) effects an extraction of certain oil constituents from the paraffinic raffinate, thus producing a fraction which is especially suited for reintroduction into the main extraction system without disturbing the desired equilibrium conditions.

While the system as a whole is particularly effective, since the lower layer thus produced in separator 21 is well adapted for reuse in the main extraction system, it will be understood that advantageous results may also be secured even if the separator 21 be by-passed, and the lower layer in separator 32 is returned to a point in the main extraction system where it does not produce the disadvantages mentioned.

Through the present development there is thus provided a process for not only recovering most of the naphthenic solvent from the paraffinic raffinate in a simple and expeditious manner, while at the same time a fraction is produced which is well adapted for reintroduction into the main counter-current extraction system without adversely affecting the settling rates of the process or causing the efficiency of extraction to be reduced.

The terms and expressions which have been

employed are used as terms of description and not of limitation, and there is no intention, in the use of such terms and expressions, of excluding any equivalents of the features shown and described, or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed.

What is claimed is:

1. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, and introducing the lower of said layers to said mixture of oil and solvents at a point between the point where the mineral oil is charged and the point where fresh maphthenic solvent is introduced.

2. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, and introducing the lower of said layers to said mixture of oil and solvents at a point where the composition of the mixture is substantially like the composition of said lower layer.

3. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, the solvents being counterflowed and the mineral oil to be treated being introduced thereto at a point intermediate the points at which the solvents are introduced, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, and introducing the lower of said layers to said mixture of oil and solvents at a point where the composition of the mixture is substantially like the composition of said lower layer.

4. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, removing at least a part of the paraffinic solvent from the upper of said layers, reducing the temperature of the balance of said upper layer to cause it to separate into upper and lower layers, recycling the latter of said lower layers to raffinate separated from the oil, and returning the first of said lower layers to the mixture of solvents and oil.

5. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, removing at least a part of the paraffinic solvent from the upper of said layers, reducing the temperature of the balance of said upper layer to cause it to separate into upper and lower layers, recycling the latter of said lower layers to raffinate separated from the oil, and returning the first of said lower layers to the mixture of solvents and oil at a point where the mixture is of substantially the same composition as the first of said lower layers.

6. In the process of extracting a mineral oil by means of paraffinic and naphthenic solvents jointly employed, said solvents comprising primarily light hydrocarbons and cresylic acid, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, and introducing the lower of said layers to said mixture of oil and solvents at a point between the point where the mineral oil is charged and the point where fresh naphthenic solvent is introduced.

7. In the process of extracting a mineral oil by means of propane and a solvent having the solvent characteristics of cresylic acid, jointly employed, the steps comprising separating a paraffinic type raffinate from the mixture of oil and solvents, causing said raffinate to separate into layers, and introducing the lower of said layers to said mixture of oil and solvents at a point between the point where the mineral oil is charged and the point where fresh naphthenic solvent is introduced.

8. A process for the separation of paraffinic and non paraffinic fractions from an oil containing the same which comprises countercurrently extracting said oil with a selective solvent and thereby forming a raffinate phase and an extract phase, separating said phases, extracting said raffinate phase with a selective solvent to produce a final raffinate phase and an intermediate extract phase and introducing said intermediate extract phase into said first mentioned extraction at a point intermediate the points of introduction of selective solvent and oil entering the system.

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