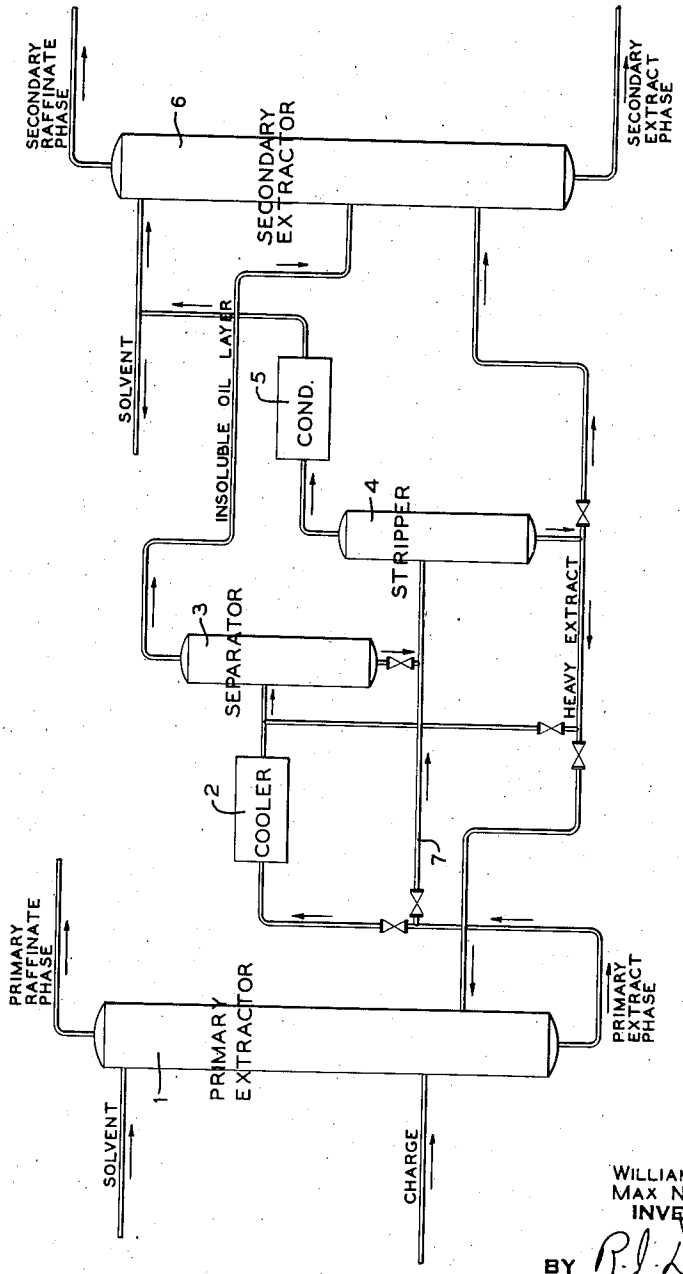
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SOLVENT REFINING OF OIL

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SOLVENT REFINING OF OIL

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This invention relates to a method of solvent refining hydrocarbon oil and has particular reference to the refining of mineral lubricating oil.

The invention contemplates the solvent extraction of oil with a selective solvent, such as furfural, to produce a primary raffinate phase containing relatively high viscosity index oil, and a primary extract phase containing oil comprising constituents of low and intermediate viscosity index. The primary extract and raffinate phases are separated and the solvent removed therefrom. The extract phase, after a large portion, or substantially all, of the solvent is removed is subjected to further extraction in a secondary extraction zone with a smaller proportion of the selective solvent in order to separate it into secondary extract and raffinate phases. The secondary raffinate phase contains oil of intermediate character, while the secondary extract phase contains oil of relatively low viscosity index.

A desirable method of proceeding in accordance with the invention involves cooling the primary extract phase after its removal from the primary extraction zone to a temperature below that prevailing in the primary extraction zone so as to separate therefrom an insoluble oil layer. The solvent is then stripped from the remaining solvent layer, all or in part, leaving a heavy extract. The insoluble oil layer is subjected to countercurrent extraction in the secondary extraction zone with a portion of the solvent removed from the primary extract phase. The heavy extract phase, from which the solvent was stripped, is introduced to the secondary extraction zone so as to enrich the secondary extract phase in low viscosity index constituents, thereby displacing relatively paraffinic constituents from the secondary extract phase which would otherwise be retained in the secondary extract phase, as withdrawn from the secondary extraction tower.

If desired, a portion of the heavy extract from which the solvent has been stripped may be introduced to the primary extraction zone where it likewise serves the purpose of enriching the primary extract phase, thereby displacing relatively paraffinic constituents which might otherwise be retained in the primary extract phase, as removed from the primary extraction zone.

A further modification of the process involves incorporating a portion of this heavy extract phase, from which the solvent has been stripped, in the primary extraction phase, after the removal of the latter from the primary extraction tower, and prior to subjecting the primary extract phase to settling.

The heavy extract material thus incorporated in the primary extract phase exerts a "salting-out" effect such that a greater proportion of oil of intermediate character is separated as an insoluble fraction upon standing at reduced temperature. A higher yield of separated oil is thus obtained than when the primary extract phase is subjected merely to cooling and settling in the absence of the added heavy extract material.

While furfural has been mentioned, it is contemplated that other solvents, having selective action as between constituents of the oil of differing viscosity indices, may be used as, for example, nitrobenzene, benzaldehyde, aniline, etc. The selective solvent may be used alone, or in the presence of a suitable auxiliary or modifying solvent as, for example, benzol and its homologs; light petroleum hydrocarbons, such as propane, gasoline or naphtha; and other solvents, such as low-boiling aliphatic ethers.

The invention contemplates producing in the primary extraction a fraction of oil of relatively high viscosity index, preferably having a viscosity index of around 100; and in the secondary extraction the production of a substantial amount of oil of intermediate character having a viscosity index, for example, of around 75.

The remaining oil contained in the secondary extraction phase is of relatively low viscosity index. It may, after removal of the solvent, be used as an inferior grade of lubricating oil, or may be disposed of in other ways, such as cracking stock for the production of motor fuel.

In order to facilitate description of the process of this invention, reference will now be made to the accompanying drawing.

As shown in the drawing, the charge oil, comprising a lubricating oil stock, is obtained from a source not shown and introduced to the mid-portion of a primary extraction tower 1. The selective solvent obtained from a source not shown is also introduced to the upper portion of this extraction tower. The oil and solvent flow countercurrently to each other through the upper portion of the tower. Complete mixing and contact between the oil and solvent may be facilitated by suitable packing material provided within the tower. Where furfural is used as the selective solvent, it is advantageous to maintain a relatively high temperature within the extraction tower. The temperature at the top of the tower may be as high as 300° F., while that at the bottom of the tower may be as high as 200° F. It is desirable to maintain a temperature differential of around 60° or more throughout the extraction zone.

These temperature conditions may be maintained by heating both the solvent and the oil to the necessary temperatures prior to introduction to the tower.

The solvent is introduced to the tower advantageously in the proportion of about five, and in some instances as much as eight, parts of solvent to one part of charge oil, where it is desired to

produce a primary raffinate of quite high viscosity index oil.

As a result of the contact between solvent and oil within the tower, primary extract and raffinate phases are produced. The raffinate phase, where furfural is the selective solvent, accumulates in the upper portion of the tower and is removed therefrom. This raffinate phase, under the temperature conditions and the solvent ratio specified above, will comprise relatively high viscosity index oil, that is, oil having a viscosity index of around 100.

The primary extract phase withdrawn from the bottom of the primary extractor is conducted through a cooler 2 wherein it is cooled to a lower temperature, as for example, in the range 130 to 160° F. The cooled material is then introduced to a separator 3 wherein separation into fractions or layers occurs. The upper layer comprises an oil of intermediate character insoluble in the solvent, while the bottom layer comprises heavy extract oil dissolved in a large body of solvent.

The heavy extract layer is withdrawn from the bottom of the separator 3, and conducted to a stripper 4, wherein all or the major portion of the solvent is stripped and removed from the heavy extract. The solvent is removed as a vapor and condensed in a condenser 5. A portion of the condensed solvent is returned to the upper portion of the secondary extraction tower 6, while that portion not so returned may be conducted to tankage for further use in the primary extractor

The insoluble oil accumulating in the upper part of the separator 3 is withdrawn and introduced to the mid-portion of the extractor 6. It is here brought into countercurrent contact with the solvent liquid being introduced to the top of the secondary extractor.

A portion of the heavy extract from which the solvent has been stripped in the stripper 4 is introduced to the lower portion of the secondary extractor 6. In this way, the solvent-oil mixture accumulating in the lower portion of the extractor 6 is enriched with heavy extract which results in displacing a substantial amount of the more paraffinic constituents retained in the solvent-oil mixture descending in the extractor 6. The paraffinic constituents so displaced are thus caused to accumulate in the upper portion of the extractor and are removed as a component of the secondary raffinate phase.

The secondary extract phase withdrawn from the bottom of the extractor 6 will thus comprise oil of relatively low viscosity index.

A portion of the heavy extract from which the solvent has been stripped in the stripper 4 is advantageously mixed with the primary extract phase prior to introduction to the cooler 2, for the purpose of aiding and increasing the amount of oil separated in the separator 3.

If desired, some of the heavy extract material may be returned to the bottom of the primary extraction tower, as indicated, for the purpose of favorably affecting the distribution of high and low viscosity index constituents of the oil between the phases formed therein.

In some cases, it may be desired to omit the step of subjecting the primary extract phase to cooling and settling. In that case, the primary extract phase is conducted through the connection 7 directly to the stripper 4 wherein substantially all the solvent is removed. All the stripped extract phase is then introduced to the lower portion of the extractor 6, as indicted, and there subjected to countercurrent extraction with a portion of the selective solvent which was stripped from it in the stripper 4.

The ratio of solvent introduced to the secondary extractor 6 may be in the proportion of about 2½ parts of solvent to 1 part of extract phase material charged to this extractor. The temperature maintained at the top of the tower may be around 250° or lower while that at the bottom of the tower is around 140° or lower. Under these conditions the secondary raffinate phase, removed from the secondary extractor, will comprise oil which, after the solvent is removed, will have a viscosity index of around 75.

The following data are indicative of the results obtained by extracting lubricating oil stock of mixed-base origin in the manner described above, and using the temperatures and solvent proportions specified.

The lubricating oil stock in question was subjected to countercurrent extraction with furfural in the primary extraction stage. The solvent was removed from the resulting primary raffinate phase, and the oil then dewaxed to produce a dewaxed primary raffinate oil amounting to about 44.8% by volume of the charge. The comparative laboratory tests on the charge and raffinate oil, before and after dewaxing, were as follows:

	Charge	Primary raffinate after removal of the solvent	Dewaxed primary raffinate oil
Gravity A. P. I.	24.0	31.6	30.7
S. U. viscosity—			
At 100° F.	1220	515	663
At 210° F.	85	66.4	72.8
Color tag rob	½	9¾	10
Pour ° F.	115	115	0
Percent carbon residue	0.92	0.03	0.02
Viscosity index	66	105	99
Vis. grav. cons.	0.838	0.788	0.792

The solvent was removed from the primary extract phase, and the extract oil remaining subjected to extraction with a smaller proportion of furfural at lower temperatures, as previously described, to produce secondary raffinate and extract phases. The solvent was removed from these phases. Secondary raffinate oil was dewaxed to produce a dewaxed oil amounting to about 25.2% by volume of the original charge.

The comparative tests on the primary extract, the secondary extract, and the secondary raffinate, before and after dewaxing, and with the solvent removed, were as follows:

	Primary extract	Secondary extract	Secondary raffinate	Dewaxed secondary raffinate
Gravity A. P. I.	16.5	9.0	26.5	
S. U. Viscosity—				
At 100° F.	5250		850	1089
At 210° F.	151	366.4	73.9	82.7
Color tag rob	Black	Black	3½	2¼
Pour ° F.	85		115	
Percent carbon residue	3.3		0.13	
Viscosity index	21		74.5	73
Vis. grav. cons.	0.888	0.943	0.822	

As a result of the foregoing extraction, a yield of 44.8% of primary raffinate, having a viscosity index of 99 after dewaxing, was obtained on the basis of the raw stock charged to the primary extraction tower. A yield of 25.2% of secondary raffinate oil, having a viscosity index of 73 after dewaxing, was obtained on the basis of the raw distillate charged to the primary extraction tower. The combined yield of raffinate oil was,

therefore, around 70% of the raw wax distillate charge.

It is recognized that heretofore it has been proposed to extract in stages in which the phases from the primary stage have been subjected to cooling and settling, and the resulting separated material subjected to further extraction, or returned to the primary extraction zone.

A feature of the present invention, however, involves removing substantially all of the solvent from the primary extract phase, and re-extracting the extract phase oil remaining after the solvent has been stripped from it in a secondary extraction zone with a relatively smaller proportion of the same solvent under the same or somewhat different temperature conditions.

The present invention involves using a relatively large proportion of selective solvent in the primary extraction step so as to produce a lubricating oil fraction of very high viscosity index, namely, around 100. The resulting extract phase, therefore, contains a large proportion of solvent which is advantageously removed, in whole or major part, so that the entire extract can be efficiently treated in the second operation with a controlled smaller quantity of solvent applied countercurrently to produce a larger yield of the intermediate grade of oil.

The present invention, therefore, comprises a process adapted to produce, by solvent extraction methods, a lubricating oil of very high viscosity index as well as a high yield of oil of intermediate character having a higher viscosity index than the charge.

It is, of course, contemplated that the solvent dosages and the extraction temperatures may vary from those set forth in the above described specific examples dependent upon the nature of the oil being treated and the degree of extraction desired.

Obviously, many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. The method of solvent refining hydrocarbon oil which comprises extracting the oil with a relatively large proportion of a selective solvent at an elevated temperature of around 200 to 300° F., forming a primary raffinate phase consisting essentially of oil having a high viscosity index of around 100 and a primary extract phase comprising oil of low and intermediate viscosity index dissolved in the bulk of the solvent, removing said primary phases, reducing the temperature of the primary extract phase to around 130 to 160° F. to separate it into an insoluble oil fraction and a heavy extract fraction containing the bulk of the solvent, stripping substantially all of the solvent from said heavy extract fraction, incorporating a portion of the stripped heavy extract in the primary extract phase after removal from the primary stage to facilitate its separation into fractions, subjecting the insoluble oil fraction to countercurrent extraction with a further quantity of selective solvent, in relatively smaller proportion than that used in the primary stage in a secondary extraction zone in the absence of feed oil, maintaining said secondary extraction zone at a temperature of around 140 to 250° F., introducing a portion of the stripped heavy extract fraction to the secondary extraction zone, forming secondary extract and raffinate phases, and removing said phases.

2. The method of solvent refining mixed base mineral lubricating oil which comprises introducing the oil to the mid-portion of a primary extraction tower, introducing furfural to the upper portion thereof in relatively large proportion, maintaining the tower at a temperature of around 200 to 300° F., forming a primary raffinate phase consisting essentially of constituents having a high viscosity index of around 100 and a primary extract phase comprising oil of low and intermediate viscosity index dissolved in the bulk of the solvent, removing said phases, reducing the temperature of the primary extract phase to around 130° to 160° F. to separate it into an insoluble oil fraction and a heavy extract fraction containing the bulk of the solvent, stripping substantially all of the solvent from the heavy extract fraction, introducing the insoluble oil fraction to the mid-portion of a secondary extraction tower wherein it is extracted in the absence of feed oil, introducing furfural to the upper portion of said secondary tower in relatively smaller proportion than that used in the primary stage, maintaining the tower at a temperature of around 140 to 250° F., introducing a portion of the stripped heavy extract fraction to the lower portion of said secondary tower, forming a secondary raffinate comprising oil of intermediate viscosity index and a secondary extract phase rich in low viscosity index constituents, and removing said phases.

3. The method according to claim 2 in which a portion of the stripped heavy extract is incorporated in the primary extract phase after removal from the primary stage to facilitate its separation into fractions.

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