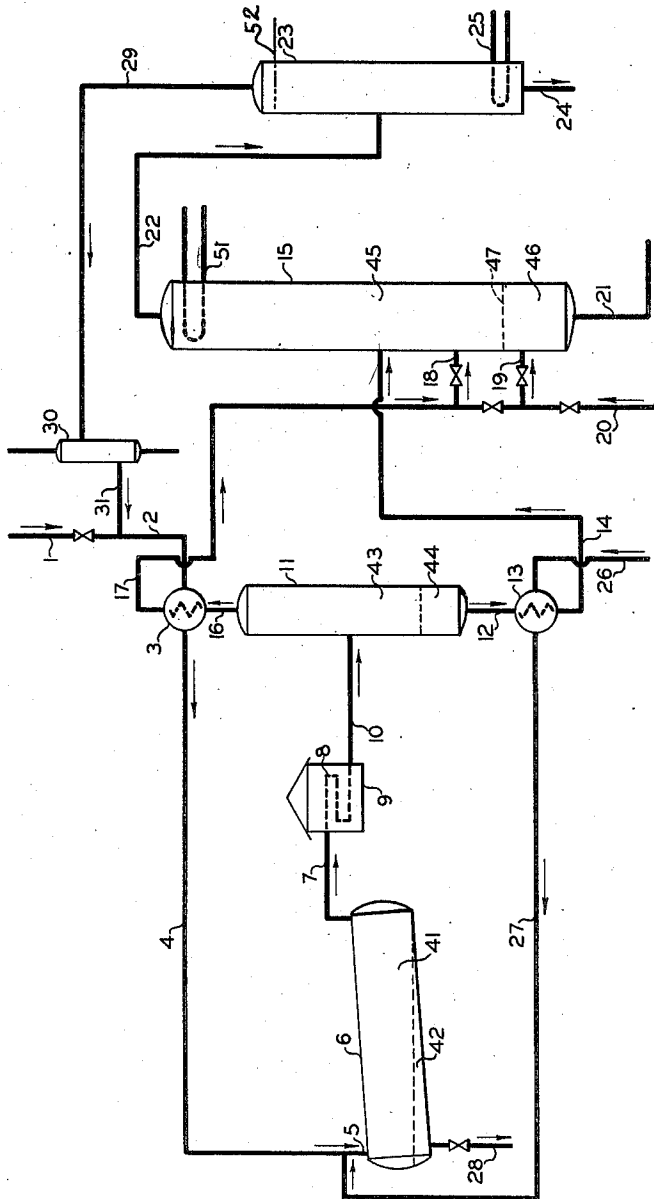


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PROCESS FOR THE PROPANE FRACTIONATION
OF LUBRICATING OIL STOCKS
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PROCESS FOR THE PROPANE FRACTIONATION OF LUBRICATING OIL STOCKS

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7 Claims. (Cl. 196—14.46)

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This invention relates to the propane fractionation of lubricating oil stocks. In one aspect it relates to a process for the propane fractionation of asphalt-containing lubricating oil stocks. In another aspect it relates to a method for the propane fractionation of asphalt-containing lubricating oil stocks wherein the process is one of continuous operation and without plugging of the fractionating column with solid or semi-solid asphaltic material.

The asphalt content of a crude oil may be defined as the per cent of residue from the vacuum distillation of a crude oil having a maximum A. S. T. M. D234-36 penetration of 100 at 77° F. Some asphalt-containing crude oil stocks contain as little as 2 per cent by weight or even less asphalt. Many asphaltic crude oils, such as California crudes, contain from 12 per cent to 65 per cent asphalt, and Wyoming and Arkansas crude oils contain about 30 per cent asphalt. It will be obvious that crude oils which contain this relatively high percentage of asphalt will produce reduced crude oil stocks containing much higher percentages of asphalt. The reduction of crude oils, of course, consists in the removal of dissolved hydrocarbon gases, gasoline, kerosene and distillate fractions, leaving as a residue the lubricating constituents with or without asphaltic constituents.

The high asphalt content crude oil stocks must be treated for removal of the asphalt content before attempting separation of the lubricating oil constituents by fractionation with propane. Even some low asphalt content crude oil stocks are not amenable to propane fractionation because of deposition of asphaltic materials in the propane fractionation column. Thus a crude oil stock containing any amount of asphaltic material may need to be treated prior to fractionation with propane in order to prevent plugging of the fractionating column with asphalt.

An object of this invention is to provide a continuous process for the fractionation of lubricating oil stocks with liquid propane.

Another object of this invention is to provide a process for the fractionation of asphaltic-containing lubricating oil stocks wherein asphaltic material is not deposited in a propane fractionating column.

Still another object of my invention is to provide a combination process for the removal of asphaltic material from asphalt-containing crude lubricating oil stocks and the subsequent separation of lubricating oil fractions with liquid propane.

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Still other objects and advantages of my process will be obvious to those skilled in the art upon reading the following description which, taken with the attached drawing, forms a part of this specification.

The drawing represents diagrammatically one form of apparatus in which to practice the process of my invention.

The separation of liquid asphaltic materials from petroleum oils and more particularly from reduced crude oils by means of single-stage extraction with propane is old in the art. Such operations are usually carried out at moderate temperatures in the range of about 70° to 160° F. in the case of Mid-Continent residua to yield asphaltic fractions of varying amounts depending on asphaltene content, temperature and propane/oil ratio employed. In addition to this conventional de-asphalting type of operation, it has been recognized for many years that a fractionating effect can be realized in the case of propane-oil mixtures subjected to temperatures in the region of 140° F. up to the critical temperature of propane. This fractionation effect is a direct result of the unique properties of propane-oil systems wherein high molecular weight components of the oil and the most aromatic are rejected from solution with increasing temperature. Thus in countercurrent towers using a stripping section and a temperature differential in the rectifying section, an effect equivalent to distillation and solvent extraction may be achieved. However, due to difficulties imposed by operating in close proximity to the critical temperature of propane, commercial exploitation of this principle in the production of the relatively light S. A. E. 30 to 50 grades of oil was unknown until comparatively recent times.

Inasmuch as the fractionation of residual stocks into close-cut fractions in the SAE 30 to 50 grades has certain obvious technical and commercial advantages, extensive investigation of this type of separation has disclosed problems not hitherto reported in the prior art. The most troublesome problem is that presented by the separation of an asphaltic phase in the countercurrent tower which renders the process inoperative in the case of high-asphalt stocks and in many representative low-asphalt mid-continent residual stocks. In the propane fractionation of three well-known and representative reduced crudes from widely separated Oklahoma fields, two were found to be unsuitable to this type of fractionation because of deposition of solid asphaltic plugs in the column. In general, each

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reduced crude oil feed stock was subjected to the following fractionation conditions to produce SAE 40 oils during the operative periods: propane/oil volume ratio, 7/1; top tower temperature, about 195° F.; bottom tower temperature, about 175° F. In the cases of the two intractable stocks the asphalt deposits built up throughout the entire length of the stripping section until virtually complete closure of the column occurred.

The exact mechanism involved in the formation of asphaltic plugs from these low asphalt stocks is not understood, nor can the usual inspection tests be relied upon to indicate which crudes are amenable to this type of fractionation. The evidence strongly indicates that in the case of certain Mid-Continent residue three phases are formed in the vicinity of the feed plate, (1) a propane-rich light oil fraction, (2) an oil-rich propane-oil solution, and (3) a semi-solid asphalt-oil solution. These facts have been verified by equilibrium bomb experiments carried out at feed plate conditions, which experiments are the only reliable index as to the amenability of a given reduced crude to propane fractionation. The actual quantity of oil-free asphaltenes involved in inoperative stocks is very small, being of the order of 0.05 to 1.0 per cent by weight as determined in the equilibrium bomb. The seriousness of the problem is further emphasized by the fact that the oil-free asphaltenes found in a completely plugged column amounted to only 10 per cent of the available free asphalt formed at the feed plate during the operating period. This apparent paradox is resolved on consideration of the fact that the solid plug contains from 50 to 80 per cent oil, thus greatly increasing the bulk of the asphaltic material. It has further been found that in the case of troublesome stocks, variations in temperature, pressure, and propane/oil ratio do not materially influence the phase relationships previously set forth.

I have now discovered a procedure whereby all crudes regardless of type can be rendered adaptable to propane fractionation. My process involves de-asphaltizing by dilution of the reduced crude with 2 to 10 volumes of propane at temperatures of from 75° F. to 160° F. and separating an asphalt and an oil phase as in conventional de-asphalting of such stocks. The relatively large amount of propane contained in the de-asphaltized oil is deleterious to the subsequent fractionating step and must be removed. Conventional removal of excess propane is carried out by distillation which is a costly procedure involving stripping columns, condensers, heaters, compressors and the like. My process entails heating of the de-asphaltized oil-propane solution, as it comes from the asphalt settler, to near or above the critical temperature of propane. Under these conditions a separation of the mixture into two phases occurs, resulting in a substantially complete separation into oil-free propane as one phase and an oil-rich phase of greatly lowered propane content as the other phase. The large difference in density of the two resulting phases promotes easy and rapid separation. The only work needed on this recovery system is the relatively small quantity of heat required to raise the temperature of the de-asphaltized oil-propane mixture to the separation temperature. Since in any case the feed to the fractionator must be heated to approximately 180° F. if an overhead SAE 40 to 50 oil is desired, the additional heat required for the sep-

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aration is merely that required to increase the temperature from about 180° F. to the range of 205° to 225° F. Furthermore, this extra heat can be recovered efficiently by means of heat exchangers. The propane recovered from the separation process is charged to the fractionating column at the conventional point admixed with fresh makeup propane or at a point between the oil and propane inlets.

Referring now to the drawing, reference numeral 6 identifies a settling tank which is tilted slightly from horizontal. This settler 6 is equipped with several inlet and outlet connections. Pipe 28 is for withdrawal of material from the bottom of the settler. Pipe 7 is for withdrawal of material from the top of the settler, while inlet 5 is a mixing device of the type of a mixing orifice or a vertical mixer or any other type of mixer which is adapted to mixing of liquid propane and an asphalt-containing lubricating oil stock at the point of introduction of the mixture into the settler tank 6. Reference numeral 8 refers to a heating coil disposed in the heater box 9. This box 9, of course, may be a fire box chamber lined with fire brick or suitable refractory to withstand any temperatures for applying heat to the coil 8. The coil heater assembly 8-9 may alternately be a heat exchanger in which superheated steam is passed in indirect heat exchange with the material to be heated.

Vessel 11 is an elongated, vertically disposed vessel used for the separation of two phases. Heat exchanger 3 is provided for exchanging of heat between material leaving the top of vessel 11 through a line 16 and material introduced into the system through some lines 1 and 2. Material exiting from the exchanger 3 may pass through line 4 for passage into the mixing device 5. Settler 11 is provided with a bottoms draw-off line 12 and this line 12 is connected with a heat exchanger 13 similar to exchanger 3. Material from the bottom of vessel 11 is intended to enter exchanger 13 through line 12 and to leave through a line 14 while material passed in heat exchange enters the system through a line 26 and leaves the exchanger through a line 27.

A vessel 15 is a fractionating vessel in which the lubricating oil stock may be fractionated with liquid propane under high pressures. This vessel is equipped with an overhead product withdrawal line 22, a bottoms withdrawal line 21, and several side inlet lines. Line 14, previously mentioned, is connected with about the center vertically of the vessel 15, while inlet lines 18 and 19 are connected with vessel 15 at progressively lower points.

Vessel 23 is a fractionating tower having an overhead vapor product withdrawal line 29, a side inlet line which connects with the overhead line 22 from vessel 15, and a bottom withdrawal line 24. A reboiler coil 25 is provided for the reboiler section of this still. Line 52 is for introduction of reflux from any source desired.

A condenser 30 is connected with the end of line 29 for the condensation of material passing from line 29 into a line 31.

A line 10 is connected with the outlet from the heating coil 8 to an inlet point in about the middle of the settler 11. A pipe 17 is provided for passage of one of the materials issuing from the heat exchanger 3 to lines 18 and 19. Line 20 is for passage of material from a source, not shown, into lines 19 and 18.

In the operation of my process, an asphalt-containing reduced crude lubricating oil stock from a source not shown enters the system through the line 26 and is heated in heat exchanger 13. This heated oil then passes on through the line 27 to the mixer 5. Liquid propane, either recycled propane as subsequently produced, or fresh propane, or a mixture of the two, is passed through the line 2 and through the heat exchanger 3 in which the charge propane is heated. The heated propane from the exchanger 3 is conducted through the pipe 4 to the mixer 5. This mixer 5 is intended to mix thoroughly and completely the propane from line 4 and the asphalt-containing oil stock from line 27, and to discharge the mixture into the settler 6. This mixer 5, as mentioned before, may be any type of mixer suitable for mixing such materials. The mixer may be installed and operated in such a manner as to discharge the mixture into the settler tank 6, or the mixer 5 may be installed within the settler tank 6, if desired. According to my process, I mix one volume of asphalt-containing crude oil stock with from 2-10 volumes of propane at a temperature between about 75° F. and 160° F. The pressure maintained in this tank is, of course, sufficiently high to make certain that the propane is a liquid under the temperature conditions mentioned. This pressure may be from about 580 lbs. to 650 p. s. i. g. Under these operating conditions, an asphaltic oil settles to the bottom of the vessel 6, while the upper propane layer 41 contains the desired lubricating oily constituents, along with undesired oily constituents. The asphaltic constituents, however, are well segregated in the asphaltic layer 42. The liquid asphalt-containing layer 42 is continuously withdrawn through the line 28 to such disposal as desired. This disposal may include a propane separation apparatus in which propane dissolved in the asphaltic material is separated by flashing or by distillation, condensed and recycled into the system. Propane from this source for recycling may be introduced into the system from pipe 1 for passage through the mixer 5, or may be introduced into the system through the line 20 for passage through pipes 19 or 18 into the fractionator vessel 15.

The propane-oil phase 41 is removed from the settler 6 through the line 7 and is conducted to the heating coil 8. Heated material from coil 8 is passed through line 10 into about a mid-point of the settler 11. In heating coil 8, the propane-oil solution is heated to a temperature of from 205-225° F. At this relatively high temperature, most of the lubricating oil stock is thrown out of solution in propane and separates as a separate phase. This phase, which is specifically heavier than the propane phase, settles to the bottom of the vessel 11 and accumulates therein as a liquid phase, identified by reference numeral 44. This liquid phase is accordingly an oily phase containing only a relatively small amount of dissolved propane, i. e., from 0.2-0.6 volume of propane per volume of oil. Under the conditions maintained in the separator 11, the lighter and upper propane phase containing a small amount of paraffinic oil in solution is identified by reference numeral 43. Under the conditions maintained in the separator 11, the propane phase 43 contains about 0.5 per cent of lubricating oil constituents by volume.

The propane containing dissolved oil removed from the top of the asphalt settler 6 contains from about 5 to 6 volumes of propane per volume

of oil and, of course, material of this composition is the charge to the settler 11.

The propane-rich phase 43 from separator 11 is removed from this vessel through the overhead line 16 to the heat exchanger 3, in which the propane product is cooled. The cooled propane solution containing the small amount of oil is passed on through the pipe 17 and is introduced into the fractionator 15 through the side inlet pipe 19. The oil-rich phase accumulating in the bottom of the separator vessel 11 is removed through the pipe 12 and cooled in the exchanger 13. The cooled oil is passed on through pipe 14 and introduced into the fractionator vessel 15 at about a mid-point vertically. The phases are separated in separator 11, prior to introduction into fractionator 15 in order to maintain a proper propane to oil ratio at the different points in the fractionator column. An oil-rich phase 46 is maintained in the bottom of this fractionator vessel 15, while a propane-rich phase is maintained as an upper layer 45. This upper propane layer 45 contains the paraffinic and more desirable lubricating oil constituents which are stripped from the oil phase 46 by propane introduced through line 19. In the vessel 15 and connected to the end of line 19 is a distributor ring or other means for providing a large surface of contact between the introduced propane and the oil phase 46. Some oily material of course is dissolved from the oil introduced through line 14 as the oil settles in countercurrent relation to the upward-rising propane. Reference numeral 47 is intended to indicate the liquid-liquid interface between the upper propane-rich layer 45, and the bottom oil-rich layer 46. The section of the vessel below the inlet 14 may be termed the stripping section, while the section of the vessel above inlet 14 may be termed the fractionating zone. The propane containing the small amount of dissolved oil introduced into the vessel 15 through line 19 is introduced at such a temperature that the section of the fractionator below the interface 47 may be maintained at a temperature between the limits of about 140° and 180° F. The charge oil introduced into the fractionator through pipe 14 is introduced at such a temperature that at its point of addition the contents of the tower are maintained at a temperature between limits of 160 and 190° F. Means for heating the top of the vessel to a still higher temperature is represented by a heating coil 51 positioned in the top of the tower. A heat exchange material is passed through the coil 51 at such a temperature and in such an amount as to maintain a temperature in the top of the fractionator between the limits of 175° and 200° F. The pressure in fractionator 15 is usually about 580-650 p. s. i. At higher temperatures the more naphthenic and aromatic, and the higher molecular weight paraffinic constituents are precipitated from solution in propane. Thus by maintaining the propane solution at the upper outlet end of the fractionator at a desired temperature, the propane solution will contain desired lubricating oil constituents. The propane-oil product leaving vessel 15 through line 22 is conducted into fractionator vessel 23. In the fractionator 23 sufficient reboil heat is supplied through the reboiler 25 to separate the propane from the lubricating oil constituents at a lower pressure than that in fractionator 15. The lubricating oil constituents are accordingly removed from the bottom of the fractionator through the line 24 for such subsequent treatment or disposal as desired. The va-

porous propane is removed through the overhead vapor line 29, is condensed in condenser 30, and the condensate passed through line 31 into line 2 as recycled propane. New or fresh propane, needed for make-up purposes or for charging the propane-to-oil ratio in the system, may be at least in part added through line 1. Additional propane for make-up purposes and especially as required for direct use in the fractionator 15 is introduced through the line 20 for addition to the propane passing from line 17 into the line 19. If desired, the propane from line 17 may be introduced into the fractionator 15 in the fractionating section through the inlet pipe 18 in place of through line 19 into the stripper section. If desired, the propane from line 17 may be introduced into the fractionator through lines 18 and 19. Ordinary pressure in the asphalt settler 6 may be somewhat higher than in subsequent apparatus along the route of travel of the propane solution, but only sufficiently higher as to cause the material to flow without the use of pumps from the settler 6 through the coil 8, settler 11, and into and out of the fractionator 15.

An overall propane-to-oil ratio is maintained between the limits of 6:1 to 14:1.

Example

In one embodiment of the present invention, a Mid-Continent residuum is treated according to the process hereinabove described. Liquid propane is passed through line 2 and heated in exchanger 3 to a temperature of about 130° F. and passed through line 4 to mixer 5. The crude oil stock residuum is passed from line 26 into exchanger 13 in which it is heated to about 130° F. This heated oil is then passed through pipe 27 to mixer 5 in which the propane and residuum are thoroughly mixed. The mixture is injected into the settler 6. Approximately 10 per cent by volume settles to the bottom as layer 42. This phase, which is quite fluid and non-fouling in nature, contains approximately equal volumes of propane and oil under conditions of temperature mentioned, and under a pressure of 580-650 p. s. i. g. The asphaltic phase is continuously withdrawn for recovery of propane and asphalt. The upper propane-oil phase which contains about one volume of oil dissolved in about 5.5 volumes of propane is continuously removed from the settler, is heated to 210-220° F. in heating coil 8, and is then introduced into the propane separator 11 in which stratification into an oil-rich bottom layer and a propane-rich upper layer occurs. The oil-rich phase contains from .2 to .6 volume of propane per volume of oil, while the propane phase contains only a very small amount of oil, usually less than 0.50 per cent by volume. The propane layer is withdrawn from separator 11 and is cooled in exchanger 3 to 175° F. and is introduced through line 19 into a lower portion of the fractionator 15. The oil phase from the separator 11 is cooled in exchanger 13 to 180° F. and is introduced into the fractionator 15 at about a mid-point. The interface between the oil phase and the propane phase in the fractionator is maintained at about a relative position indicated in the drawing. To produce an overhead oil of 78 to 82 S. U. S. viscosity at 210° F., the fractionator is operated under the following conditions: top lower temperature, 195° F.; feed plate temperature 180° F.; and bottom temperature, 175° F. An overall propane-to-oil ratio of 8:1 is maintained, and the pressure is held constant for the entire system at 640

p. s. i. g. When a pressure of 640 lbs. is maintained in the fractionator 15, pressures at upstream points are slightly higher to overcome resistance of fluid flow. The overall propane-to-oil ratio mentioned takes into consideration the propane contained in the oil feed entering at the feed plate, as well as makeup propane from line 20, and the propane from the top of the separator 11.

If it is desired to operate the fractionator 15 at a slightly lower pressure, slightly lower temperatures in the column may be used. However, sufficient pressure must be used at all times to maintain the entire contents of the fractionator in the liquid or dense phase.

In conventional processes involving propane de-asphalting and propane fractionation, propane is separated from the asphalt-free propane-oil solution by distillation. Such an operation involved the use of a fractionator, pumps, reboil heat, and an overhead vapor condenser. The use of such apparatus in a high pressure system is, of course, expensive to construct and operation costs are high. In my process, I heat the asphalt-free propane-oil solution from the propane separation temperature to a temperature between approximately 205° and 225° F., at which temperature the oil is nearly insoluble in propane. Thus, physical separation between the oil and propane is easily accomplished since there is a considerable spread in specific gravity of these two materials. While some cost is involved in heating the oil-propane mixture to this temperature, the actual cost of this operation is minimized since the propane solution and charge oil to the fractionator have to be heated to 175° to 180° F. for efficient fractionation and the production of a desired grade of oil. Thus, the net cost of effecting the phase separation between the oil and propane in the separator 11 is of heating from 175°-180° F. to 205-225° F. In actual practice, even this amount of heating is not a direct cost item since propane feed to the mixer 5 is heat exchanged with the propane phase from the separator 11, and the oil phase from the separator is likewise heat exchanged with the incoming crude residuum. Thus, my system may be operated for considerably less cost than a conventional system, as mentioned above, involving separation of the propane from the oil by distillation methods prior to the oil fractionation step.

My process is not limited to any particular type of reduced crude lubricating oil charge stock. De-asphalting conditions will, of course, vary with the type of charge oil involved. The temperature to be maintained in the phase separator 11 will, of course, be dependent upon the extent of separation of the oil from the propane desired, and likewise, the temperatures and temperature differentials to be maintained in the fractionator 15 will be dependent upon the grade of oil desired to be taken overhead. The recovery of the propane from the de-asphalted oil and its reuse in the subsequent propane fractionation step will be operative regardless of the relative volumes of propane and oil required in the asphalt removal step within the stated limits.

The oil phase containing some propane in solution issuing from the fractionator 15 through the line 21 may be conducted to a second oil fractionation step similar to that carried out in fractionator 15, but for the production of a higher viscosity grade of oil. If desired, of course, the oil phase from pipe 21 may be passed to a flash separator or a still for separation of the propane

and oil. The propane thus separated may be combined with the propane from the fractionator 23 for recycling into the asphalt settler 6. Likewise, a portion of this recycle propane may be introduced through line 20 along with makeup propane as needed. Additional propane to the system may be introduced through line 20 directly to the fractionator 15 or through line 1 directly to the asphalt settler 6, or into both the fractionator 15 and the settler 6.

Materials of construction may be selected from among those commercially available. Special corrosion resistant materials need not ordinarily be used since in the treatment of such oils corrosion problems are small. However, the apparatus of such a system will need to be constructed to withstand safely pressures of operation as hereinabove described. Such auxiliary apparatus as pumps, valves, temperature and pressure indicating and recording devices, are not shown in the drawing, nor described in the specification, for purposes of simplicity. Likewise, pressure and temperature controllers or flow controller apparatus are not shown. The installation and use of such auxiliary apparatus is well understood by those skilled in the art.

The above-described flow system and operating conditions are given for illustrative purposes and should not be regarded as limiting the invention, the scope of which is set forth in the following claims.

Having described my invention, I claim:

1. A process for the fractionation of asphalt-containing lubricating oil stocks comprising maintaining a body of propane rich in dissolved oil in a first treating zone and a body of asphaltic material containing dissolved propane below said body of propane rich in dissolved oil in said zone, said bodies meeting at a common interface, introducing propane and said asphalt-containing lubricating oil stock into said zone, removing said asphaltic material containing dissolved propane from said zone; maintaining a body of propane lean in dissolved oil in a second zone at a temperature above that maintained in said first zone and a body of an oil-rich phase containing propane below said body of propane lean in dissolved oil in said second zone at said temperature, said bodies in said second zone meeting at a common interface, removing propane rich in dissolved oil from said first zone, and heating this latter removed material and introducing same into said second zone; maintaining a body of propane containing dissolved oil in a third zone and a body of an oil-rich phase below said body of propane containing dissolved oil in said third zone at a temperature below said temperature maintained in said second zone, said bodies in said third zone meeting at a common interface, removing propane lean in dissolved oil from said second zone, cooling this latter removed material and introducing this removed and cooled material into said third zone at a point near said interface, removing oil-rich phase from said second zone and cooling this latter removed material and introducing this cooled material into the body of propane containing dissolved oil in said third zone at a point above the point of introduction of said propane lean in dissolved oil, removing oil-rich phase and propane containing dissolved oil from said third zone, and recovering oil from this latter removed oil-rich phase and oil from this latter propane containing dissolved oil as products of the process.

2. A process for the fractionation of asphalt-containing lubricating oil stocks comprising

maintaining a body of propane rich in dissolved oil in a first treating zone and a body of asphaltic material containing dissolved propane below said body of propane rich in dissolved oil in said zone, said bodies meeting at a common interface, introducing propane and said asphalt-containing lubricating oil stock into said zone, removing said asphaltic material containing dissolved propane from said zone; maintaining a body of propane lean in dissolved oil in a second zone and a body of an oil-rich phase containing propane below said body of propane lean in dissolved oil in said second zone, said bodies in said second zone meeting at a common interface, removing propane rich in dissolved oil from said first zone, and introducing same into said second zone; maintaining a body of propane containing dissolved oil in a third zone and a body of an oil-rich phase below said body of propane containing dissolved oil in said third zone, said bodies in said third zone meeting at a common interface, removing propane lean in dissolved oil from said second zone, and introducing this removed material into said third zone at a point near said interface, removing oil-rich phase from said second zone and introducing this material into the body of propane containing dissolved oil in said third zone at a point above the point of introduction of said propane lean in dissolved oil, removing oil-rich phase and propane containing dissolved oil from said third zone, and recovering oil from this latter removed oil-rich phase and oil from this latter propane containing dissolved oil as products of the process.

3. In the process of claim 1 maintaining the temperature of the liquid phases in the first zone between the limits of 75° and 160° F., the temperature of the phases in the second zone between the limits of 205° and 225° F., and the temperatures in the top, bottom, and at the oil-rich phase feed point of said third zone between the respective limits of 175° and 200° F., 140° and 180° F., and 160° and 190° F., and maintaining the pressure in the system within the limits of 580 and 650 pounds per square inch.

4. In the method of claim 3, maintaining the volume ratio of the propane to charge oil feed to the first zone between the limits of 2 to 10, and maintaining the overall volume ratio of propane to oil introduced into the third zone between the limits of 6:1 and 14:1.

5. In the method of claim 1 maintaining the temperature of the phases in the first zone at 130° F., the temperature of the phases in the second zone at 205° F., and the temperatures in the top, bottom, and oil phase feed point in the third zone at the respective temperatures of 195° F., 175° F., and 180° F., and maintaining the pressure in the system at 640 pounds per square inch gauge.

6. In the method of claim 5, maintaining the volume ratio of the propane to charge oil feed to the first zone at 5 to 1, and maintaining the overall volume ratio of propane to oil introduced into the third zone at 8 to 1.

7. In the process of claim 1 recovering the propane from the latter removed oil-rich phase, from the oil from the said last propane containing dissolved oil and from the removed asphaltic material containing dissolved propane and recycling said recovered propane to the system.

ALBERT N. DE VAULT.

No references cited.

Certificate of Correction

Patent No. 2,527,404

October 24, 1950

ALBERT N. DE VAULT

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 1, line 14, for "D234-36" read *D243-36*; column 7, line 71, for the word "lower" read *tower*; column 8, line 65, for "regardness" read *regardless*; column 10, line 30, for "cotaining" read *containing*;

and that the said Letters Patent should be read as corrected above, so that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 20th day of February, A. D. 1951.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.