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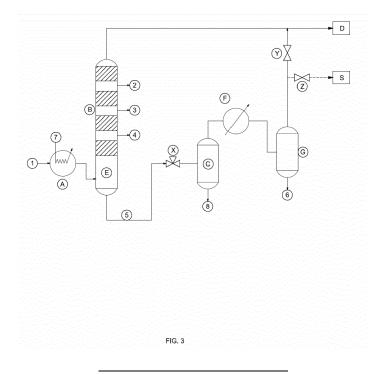
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(54) METHOD FOR INCREASING THE YIELD OF LUBRICATING BASES IN THE REGENERATION OF USED OILS

(57) The invention defines a process to rerefine used petroleum oils comprising: (a) separating water, light hydrocarbons and asphalts from used petroleum oils and obtaining a lube base oil fraction; (b) distilling the fraction obtained in step (a) in a continuous vacuum fractionation column at moderate temperatures and separating, as lateral extractions, the light, medium and part of the heavy base oils contained in said fraction and, as bottom product, a stream containing the rest of the heavy lube base

oils; and (c) evaporating the bottom product obtained in step (b) at moderate temperatures and under vacuum, the absolute pressure of the evaporation being below the absolute pressure of the flash zone in the distillation column of step (b), and obtaining heavy lube base oil vapors, that are condensed and recovered as a liquid heavy lube base oil, and a liquid asphaltic product as a bottom product.



Description

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FIELD OF THE INVENTION

[0001] The invention is related, in general, with used oil rerefining, an industrial operation that consists in recovering the lubricant base oils, by separating them from the other products and contaminants, so that they can be reformulated and used again as lubricants.

[0002] More specifically the invention describes a method to increase the yields of the rerefining process by improving the recovery of the higher boiling, more viscous base oils contained in the used oils.

BACKGROUND OF THE INVENTION

[0003] Refined petroleum oils, which are used to manufacture lubricants and other industrial oils, are called lubricant bases, base oils, or lube oil bases. Lubricants and other industrial oils are produced by mixing lubricant bases with additives.

[0004] The oils, discarded after having been used in engines or other machines, are called used oils. They contain, lubricant bases and additives and their breakdown products (lighter petroleum fractions such as naphtha and gas-oil, and heavier ones such as asphalt and coke). They also contain contaminants acquired during their collection from garages and petrol stations, such as water, glycol and solvents.

[0005] The separation of asphalts, additives and breakdown products is usually done by vacuum distillation of the oil bases as, for example, the Vaxon process described by K. Kenton and J. Hedberg in the First International Congress of Liquid Waste Refining (May 23rd 1994, San Francisco); or WO 9407798 of Viscolube Italiana Spa (1994).

[0006] Processes which separate asphalts and additives by distillation, preferably heat the used oil at 350 °C or more, producing deteriorated lubricant bases that require a final finishing step, to achieve the colour, odour and acidity specifications of virgin lube bases. For this reason, patents NL 8304023 (KTI, 1985) or EP 574272 (Chem. Eng. Partners, 1993), among others, use catalytic hydrogenation systems, for this final finishing step.

[0007] Nevertheless, because of the large investment and operational costs required for catalytic hydrogenation, there are attempts to search for alternatives. Thus WO9421761 (Sotulub, Tunisia 1994) separates asphalts and additives in a thin film evaporation with moderate temperatures (310°C) and high vacuum (1 mbar). Snam Progetti, (BE 873451, 1979) separates asphalts at low temperature by solvent extraction and SENER (ES 2199697A1, 2003), destroys and separates the metallic additives by chemical demetallization at moderate temperatures.

[0008] Even when the processes for separating asphalt and additives are carried at moderate temperatures, the quality of the base oils does not fully reach the current levels of the virgin lube bases obtained by first refining of crude oil.

[0009] In order to further improve the quality of the base oils, avoiding the expensive catalytic hydrogenation process, the addition of small amounts of caustic or reducing chemicals has been proposed. This is the case of US 7226553B2 (Sener, June 5, 2007) or WO9826031A1 (Sotulub, Tunisia 1998) which use alkaline hydroxides or hydrazine additions in the distillation.

[0010] In conclusion, current processes of used oil rerefining, that do not use catalytic hydrogenation, consist of: i) separating the water, light hydrocarbons and asphalts by different technologies, such as liquid-liquid solvent extraction, thin film evaporation and chemical demetallization, characterized in that they work at moderate temperatures; and ii) the fraction obtained after these separations, containing the lube oil bases is then distilled in a fractionation column under vacuum and moderate temperature to separate and purify the different lube bases.

[0011] Although the vacuum systems of these processes may be designed to provide very low pressures at the head of the distillation towers and special packing or plates may be used to provide low pressure drop in the fractionating column, the operating conditions of pressure and temperatures, prevailing in the flash zone of the tower, determine that part of the heavier bases will not boil and will be lost with the bottom product, which is usually valued as a low price component of asphalt or fuel-oil. These losses of lube bases are significative when the fractionation column runs at moderate temperature (e.g. below 340°C at the flash zone) to avoid quality deterioration of the base oils; and when no steam injections are used in the distillation, to avoid producing contaminated waste water.

[0012] More important losses of heavier base oils are experimented when the used oil, to be rerefined, has been collected in regions of temperate or warm climatic conditions, where high viscosity lubricant oils are used, containing significant proportions of SN-500 and bright-stock base oils.

[0013] The recovery of these heavier, more viscous oils, from the bottom product of the fractionation tower has been tried by liquid-liquid extraction, then requiring more equipment and operational costs, economically unjustified.

⁵⁵ **[0014]** The present invention describes a simple and economic process for recovering part of the heavier lube bases that are normally lost in the rerefining operation.

SUMMARY OF THE INVENTION

[0015] The main objective of the invention is to increase the yield of lube bases, in used oil rerefining, by recovering the heavier and more viscous lube bases that are lost during the fractionation and purification of the different lube bases by distillation.

[0016] Another objective of the invention is to obtain high yields of lube bases of the same quality as the virgin bases produced by first refining of crude oil.

[0017] Another objective of the invention is to obtain high yields of high quality lube bases without the need of catalytic hydrogenation technologies.

BRIEF DESCRIPTION OF THE DRAWINGS

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Fig 1 to 6 represent the distillation and fractionation stage to separate and purify the different lube oil bases from a fraction of lube oil bases (1) obtained after separating the water, asphalt and light hydrocarbons contained in the used oils.

Fig 1 and 2 illustrate the distillation and fractionation process to separate and purify the lube bases (2) (3) and (4) according to prior art technology for rerefining used oils. Fig 1 corresponds to a fractionation tower (B) operating under vacuum (D) equipped with a tubular exchanger (A) to heat the feed (1) to the column, while Fig 2 shows a thin film reboiler (A) for the same purpose. In both cases an auxiliary thermal oil (7) is used to supply the required heat. Addition of caustic (13) is also provided before the distillation.

Fig 3, 4, 5 and 6 illustrate the process for the distillation and fractionation of the lube bases in accordance with the teachings of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

- [0019] The present invention defines a process to rerefine used petroleum oils, thereafter "the process of the invention", comprising the following steps:
 - (a) separating water, light hydrocarbons and asphalts from used petroleum oils and obtaining a lube base oil fraction;
 - (b) distilling the lube base oil fraction obtained in step (a) in a continuous vacuum fractionation column at moderate temperatures and separating, as lateral extractions, the light, medium and part of the heavy base oils contained in said fraction and, as bottom product, a stream containing the rest of the heavy lube base oils; and
 - (c) evaporating the bottom product obtained in the fractionation column of step (b) at moderate temperatures and under vacuum, the absolute pressure of the evaporation being below the absolute pressure of the flash zone in the distillation column of step (b), and obtaining heavy lube base oil vapors, that are condensed and recovered as a liquid heavy lube base oil, and a liquid asphaltic product as a bottom product.

[0020] Within the context of the invention, the term "used petroleum oils" refers to lubricant or industrial petroleum oils discarded after their use in engines or other machines, and also to other petroleum oils such as contaminated lubricant oils or waste from petroleum oils not being characterized as lubricant oils.

[0021] Within the context of the invention, the term "moderate temperatures" refers to temperatures less than 350 °C and the term "low pressure" refers to absolute pressures below 15 mbar (1.5 kPa).

[0022] As previously explained, the present invention consists of a rerefining process, characterized in that the distillation and fractionation stage to separate and purify the lube bases is carried in two interconnected steps as shown in Fig. 3.

[0023] After separating water, light hydrocarbons and asphalts, the fraction containing the lube base oils (1) is firstly fractionated in a continuous fractionation column (B) operated at low pressure provided by the vacuum system (D) and at moderate temperatures, separating, as lateral extractions, the light (2), medium (3) and part of the heavy base oils (4) contained in said fraction, and as bottom product (5) a stream containing the heavier lube base oils. In this step (b) the distillation at the flash zone of the fractionation column is preferably carried out at a temperature of 310-335 °C; being the pressure in the head preferably of 2-8 mbar (0.2-0.8 kPa) and in the flash zone of 15-20 mbar (1.5-2.0 kPa). [0024] As shown in said Fig. 3, the bottom of the distillation tower (5), containing part of the heavy lube base oils, suffers an adiabatic expansion by a pressure reduction in valve (X) and flows to the vapor-liquid separator (C), where heavy lube base oil vapors are separated from the liquid asphaltic products (8) that are obtained at the bottom of separator

(C). The heavy lube base oils vapors are condensed in cooler (F) and collected in vessel (G) as a liquid heavy lube base oil (6). The vapor-liquid separator (C) is connected to the vacuum system (D). Consequently the pressure in (C) is lower than the pressure in the flash zone (E) of column (B), thus producing an adiabatic expansion, which results in the evaporation of part of the heavy base oils contained in (5). The difference of the pressure of the flash zone (E) in column (B) in respect to the pressure in separator (C) is approximately equivalent to the pressure loss of the distillation column. [0025] Therefore in a particular embodiment of the process of the invention, the evaporation of step (c) is produced by adiabatic expansion of the bottom product of step (b) to a lower pressure.

[0026] Heavier lube bases, that do not boil at the higher pressure of the column flash zone (E), will boil in separator (C) at a lower pressure, while keeping in both places the moderate temperatures required to avoid deterioration of the lube oil base quality. Gentle heating is provided to this effect by thermal fluid (7) in preheater (A).

[0027] A different embodiment of the invention, intended to increase even more the base oil recovery, consists of providing a lower pressure in (C) by a different vacuum system (S), showed in **Fig. 3-4** in dotted lines, where the system (S) provides a lower pressure than system (D).

[0028] Therefore in a particular embodiment of the process of the invention, the lower pressure of the evaporation in step (c) is obtained with the same vacuum system employed in step (b).

[0029] In another particular embodiment of the process of the invention, the lower pressure of the evaporation in step (c) is obtained by a vacuum system supplying a lower pressure than that supplied by the vacuum system of step (b).

[0030] A different embodiment of the invention is shown in **Fig. 4** where the vapor-liquid separator (C) of Fig. 3 has been substituted by a thin film evaporator (C). This thin film evaporator could be replaced with any other low pressure drop evaporation equipment known in the art.

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[0031] Therefore in a particular embodiment of the process of the invention, an additional evaporation is carried out in a low pressure drop evaporation equipment such as a thin film, falling film or short path evaporator. In a preferred embodiment of the process of the invention, the additional evaporation is carried out in a thin film evaporator. In this equipment the temperature is preferably comprised between 320 and 345 °C and the pressure is preferably comprised between 2 and 8 mbar (0.2-0.8 kPa).

[0032] As shown in Fig. 4, the bottom of the distillation tower, (5) containing part of the heavy lube base oil flows to the thin film evaporator (C) through pressure reducing valve (X). The thin film evaporator (C) is connected to the vacuum system (D) and not as in prior art technology to the flash zone (E) of the column as described in Fig. 2. Consequently the pressure in the thin film evaporator (C) is lower than the pressure in the flash zone (E), since the vapors from the thin film evaporator do not travel through the packings or plates of the distillation column to the vacuum system. Again, as shown in Fig. 3, the difference of the pressure in the flash zone in respect to the pressure at the head of the thin film evaporator (C) is approximately equivalent to the pressure loss of the distillation column.

[0033] Heavier lube bases, that do not boil at the higher pressure of the column flash zone, will boil in the thin film evaporator at a lower pressure, while keeping in both places the moderate temperatures required to avoid deterioration of the lube base quality. Both the distillation column preheater (A) and the thin film evaporator (C) are heated with a thermal fluid (7). The heavy base oils vapors evaporated in film evaporator (C) are condensed in (F) and collected in (G) as liquid heavy base oils (6). The residue (8) left after evaporating the heavy lube base oils, is collected at the bottom of the evaporator (C).

[0034] Prior technology may use a thin film evaporator as a column feed preheater or as a column reboiler (see Fig. 2) but in that case the thin film evaporator supports the pressure drop of the distillation column and the heavier base oils will not distill, being lost with the bottom of the column (B).

[0035] The thin film evaporator (C) of the invention in Fig. 4 supports a lower pressure, which favours the evaporation of the heavier base oils that are consequently recovered. It has a much smaller size than the one used in prior technology, since it deals with a smaller flow rate and has to evaporate much less amount of base oil. This is important since thin film evaporators are considerably more expensive than tubular exchangers normally used as column feed preheaters or reboilers.

[0036] A different embodiment of the invention is depicted in **Fig. 5**. In this case the vacuum system (S) provides an even lower pressure to the evaporator (C), which will give more flexibility to the process since the pressure difference between the flash zone of the distillation tower and the boiling liquid at the evaporator can be made higher.

[0037] Fig 5 also illustrates another embodiment of the invention consisting of a very simple rectification device (H) introduced between the evaporator (C) and the condenser (F), having a very low pressure loss, to improve the quality of the heavier oils (6) recovered in the thin film evaporator. This simple rectification device (H) is a low pressure drop rectification device consisting of at least a distillation plate or a simple packing bed.

[0038] As previously explained, the heavy lube base oils vapors obtained in (C) are condensed in cooler (F) and collected in vessel (G) as a liquid heavy lube base oil. A part of this liquid heavy lube base oil is recirculated and put into contact in the low pressure drop rectification device (H) with the vapors exiting from (C).

[0039] Therefore, in a particular embodiment of the process of the invention, the vapors of step (c) before being condensed are contacted in a low pressure drop rectification device, consisting of at least a distillation plate or a simple

packing bed, with part of the liquid heavy base oil obtained subsequently in step (c) which is recirculated after condensation

[0040] The bottom product (8) obtained in the evaporator (C) is a high viscosity asphaltic oil and sometimes is mixed with a fluxing fluid for flowing it through the pipes. Therefore, in other particular embodiment of the process of the invention, the viscosity of the bottom product obtained in step (c) is reduced by mixing it with a fluxing fluid.

[0041] In a preferred embodiment, the fluxing fluid is a part or the total of the light fraction obtained in step (b) as first side cut of the fractionation column.

[0042] Fig. 5 also illustrates this embodiment of the invention consisting of modifying the column conditions to obtain as top column side product a heavy gas-oil and a light base oil (2), being partly (11) or totally used as a fluxing fluid to reduce the viscosity of the bottom product (8) of the evaporator (C).

[0043] An alternative to obtain a fluxing fluid from the top of column (B) is shown in **Fig 6.** The temperature at the top of column (B) is allowed to raise in such a way that the heavy gas-oil, contained in the light side cut, distills over the top of the column, is condensed in heat exchanger (K) and collected at the bottom of separator (L), as fluxing fluid to be mixed with the bottom product (8), shown in any of the Figs. 3 to 5.

[0044] Then, in another preferred embodiment of the process of the invention, the fluxing fluid is the heavy gas-oil contained in the light base oil as first side cut in step (b) that is obtained by increasing the temperature at the top of the fractionation column and condensing the vapors that distill over.

[0045] The recovery of heavier oils is enhanced by reducing its partial pressure in the distillation or evaporation steps. Introducing an inert gas, such as nitrogen, in the distillation or evaporation, or in both simultaneously reduces the partial pressure of the oils, resulting in higher yields of the recovered heavy oils.

[0046] Then, in another embodiment of the process of the invention an inert gas is introduced in the distillation step (b), in the evaporation step (c) or both in the distillation step (b) and in the evaporation step (c). In a preferred embodiment of the process of the invention, nitrogen gas is introduced in the distillation step (b), in the evaporation step (c) or in both, in the distillation step (b) and in the evaporation step (c). In a more preferred embodiment of the process of the invention, nitrogen gas is introduced in the evaporation step (c).

[0047] This may be done in several places of the possible devices employed to carry out the process of the invention. The nitrogen gas can be mixed with the lube fraction (1) obtained in the step (a) before being distilled in step (b), or bubbled at the bottom of column (B) or at the bottom of separator and/or evaporator (C) in Figures 3 to 6 or simultaneously in more than one of the places indicated above.

[0048] The quality of the base oils recovered in the process of the invention can be improved when a basic compound is added. So that, in a particular embodiment of the process of the invention, the distillation of step (b), or the evaporation of step (c) or both are carried out in the presence of a basic compound. In a preferred embodiment, the basic compound is an alkaline hydroxide or a mixture of alkaline hydroxides, preferably in an amount below 12 kg per metric ton of base oil fraction fed to the distillation step (b). One of the preferred alkaline hydroxides to be used is KOH.

[0049] In step (a) of the process on the invention, the separation of water, light hydrocarbons and asphalts from used petroleum oils is carried out for obtaining a fraction that contains the lube base oils (1). These lube base oils (1) will be later separated and purified by fractionation (step b) and evaporation (step c), as previously stated.

[0050] In a particular embodiment of the process of the invention, the separation of step (a) is carried out by solvent extraction. In a preferred embodiment, the separation of step (a) is carried out by solvent extraction with liquid propane.

[0051] Water, light hydrocarbons and asphalts are separated by means of a solvent extraction, wherein the solvent rejects the water and the asphalt and dissolves the base oils and light hydrocarbons. This extract produces the lube base oil fraction after separating the solvent and the light hydrocarbons by vaporization.

[0052] In another particular embodiment of the process of the invention, the separation of step (a) is carried out by chemical demetallization.

[0053] In another particular embodiment of the process of the invention, the separation of step (a) is carried out by thin film vacuum evaporation.

[0054] The following examples are meant to illustrate, but in no way to limit, the claimed invention.

EXAMPLES

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Example 1 (Prior technology) (Fig 1)

[0055] As used oil, a product with following characteristics was used:

Specific gravity at 15 °C (ASTM D 1298) 0.898

Water content wt% (ASTM D 95) 4.2

Light hydrocarbons (up to 370°C) wt% (ASTM D 86/D1160) 5.7

(continued)

Viscosity at 40°C (ASTM D 446) cst 97.55 (97.55 10⁻⁶ m²/s) Viscosity at 100°C (STM D 446) cst 12.57 (12.57 10⁻⁶ m²/s)

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Distillation curve (ASTM D 1160)

Volume Distilled %	Temperature °C (corrected at atm. Pressure)		
IBP	227		
5	411		
10	450		
20	473		
30	495		
40	509		
50	522		
60	540		
70	553		
80	558		
P.F 82.3	561		

[0056] 1000 kg per hour of the used oil, specified before, was extracted with 2500 kg of liquid propane per hour in a continuous system in accordance with the indications of US 7226533B2 (June 5, 2007) for separating water, asphalts and light hydrocarbons.

[0057] The remaining 858 kg/h of the fraction, containing the lube bases was heated to 330°C and introduced in the fractionation column. The pressure in its head was 5 mbar (0.5 kPa) and in the flash zone 14 mbar (1.4 kPa). The yields obtained were:

Spindle oil (SN-80) 20 kg/h
Lube bases (SN150 and 350) 208 kg/h
Bottom product: 630 kg/h

[0058] The distillation was carried out in the presence of 7 kg of potassium hydroxide per hour.

[0059] The lube bases had a colour of 1.5- (ASTM D 500) and acidity of 0.01 mg KOH/g (ASTM D 6644).

[0060] This example shows that, with prior art technology, the recovery of base oils (SN 150-SN 350) from this particular viscous used oil is only 21.7% in dry weight, when the distillation is run at a moderate temperature of 330°C in the flash zone of the fractionating tower.

Example 2 (Present invention) (Fig. 3)

[0061] The same used oil as indicated in Example 1 was extracted with propane at a rate of 1000 kg/h After eliminating the water, asphalts and light hydrocarbons, 858 kg/h of a fraction containing the lube bases were obtained and heated to 330°C, before being introduced in the fractionating column. The pressure at the head of the column was 5 mbar (0.5 kPa) and 16 mbar (1.6 kPa) in the flash zone.

[0062] Following products were obtained in the fractionation column:

Heavy gas-oil: (SN-80) 20 kg/h Lube bases: (SN150 and 350) 208 kg/h Bottom product: 630 kg/h

[0063] The lube bases had a color of 1.5- and an acidity of 0.01 mg KOH/g.

[0064] The bottom product was expanded through valve (X) in Fig. 3 from 16 mbar (1.6 kPa) to about 5 mbar (0.5 kPa) in separator (C). An additional lube oil base (6) was recovered as distillate in vessel (G) at a rate of 86 kg/h, characterized as an SN-500, having a colour of 1.5+ and an acidity of 0.02 mg KOH/g. Both the distillation and the evaporation were carried out in the presence of 8 kg/h of potassium hydroxide.

[0065] This example shows that this particular heavy used oil yields 30.6% in dry weight of lube bases of high quality in the case of the invention, instead of 21.7% yield, which is obtained with the prior art technology in Example 1.

Example 3 (Present invention) (Fig.4)

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[0066] The same used oil indicated in Example 1 was extracted with propane at a rate of 1000 kg/h, as indicated in said example.

[0067] After eliminating the water, asphalts and light hydrocarbons, 858 kg/h of a fraction containing the lube bases were obtained and heated to 330°C, before being introduced in the fractionating column. The pressure at the head of the column was 5 mbar (0.5 kPa) and 16 mbar (1.6 kPa) in the flash zone.

[0068] The bottom product (5) of the distillation tower (630 kg/h) was expanded trough valve (X) and evaporated in a thin film evaporator as shown in Fig. 4 at 340°C and 3 mbars (0.3 kPa). An additional lube base oil was recovered in (G) as distillate at a rate of 377 kg/h, characterized as a SN-500, having a colour of 2.0- and an acidity of 0.03 mgr/l. Both the distillation and the evaporation were carried out in the presence of 8 kg/h of potassium hydroxide.

[0069] This example shows that this particular heavy used oil yields 60.9% in dry weight of lube bases of high quality, when processed according with the embodiment of the invention of Fig. 4, instead of the 21.7% yield which is obtained with the prior art technology depicted in fig.1 (Example 1).

Example 4 (Present invention) (Fig.3)

[0070] The same used oil indicated in example 1 was extracted with propane at a rate of 1000 kg/h, as indicated in said example.

[0071] After eliminating the water, asphalts and light hydrocarbons, 858 kg/h of a fraction containing the lube bases were obtained and heated to 330°C, before being introduced in the fractionating column. The pressure at the head of the column was 5 mbar (0.5 kPa) and 16 mbar (1.6 kPa) in the flash zone.

[0072] The bottom product (5) of the distillation tower (630 kg/h) was expanded through valve (X) to about 5 mbar (0.5 KPa) into separator (C), while bubbling nitrogen at the bottom of separator (C) at a rate of 10 Nm3/h. An additional lube oil base (6) was recovered as distillate in vessel (G) at a rate of 109 kg/h.

[0073] This example shows that reducing the partial pressure at the evaporation stage, by introducing nitrogen gas, increases the oil yield from 30.6% in dry weight of example 2 to 33.1%.

Claims

- 1. A process to rerefine used petroleum oils that comprises the following steps:
 - (a) separating water, light hydrocarbons and asphalts from used petroleum oils and obtaining a lube base oil fraction;
 - (b) distilling the lube base oil fraction obtained in step (a) in a continuous vacuum fractionation column at moderate temperatures and separating, as lateral extractions, the light, medium and part of the heavy base oils contained in said fraction and, as bottom product, a stream containing the rest of the heavy lube base oils; and (c) evaporating the bottom product obtained in the fractionation column of step (b) at moderate temperatures and under vacuum, the absolute pressure of the evaporation being below the absolute pressure of the flash zone in the distillation column of step (b), and obtaining heavy lube base oil vapors, that are condensed and recovered as a liquid heavy lube base oil, and a liquid asphaltic product as a bottom product.
 - 2. The process according to claim 1, wherein the lower pressure of the evaporation in step (c) is obtained with the same vacuum system employed in step (b).
- 3. The process according to claim 1, wherein the lower pressure of the evaporation in step (c) is obtained by a vacuum system supplying a lower pressure than that supplied by the vacuum system of step (b).
 - **4.** The process according to anyone of previous claims, wherein the evaporation of step (c) is produced by adiabatic expansion of the bottom product of step (b) to a lower pressure.
 - **5.** The process according to claim 4, wherein an additional evaporation is carried out in a low pressure drop evaporation equipment such as a thin film, falling film or short path evaporator.

- **6.** The process according to anyone of previous claims, wherein the vapors of step (c) before being condensed are contacted in a low pressure drop rectification device, consisting of at least a distillation plate or a simple packing bed, with part of the liquid heavy base oil obtained subsequently in step (c) which is recirculated after condensation.
- 7. The process according to anyone of the previous claims, wherein an inert gas is introduced in the distillation step (b), in the evaporation step (c) or in both, in the distillation step (b) and in the evaporation step (c).
 - 8. The process according to claim 7, wherein nitrogen gas is mixed with the lube oil fraction obtained in step (a) before being distilled in step (b); or is introduced at the bottom of the fractionation column of step (b); or is introduced in the separator and/or evaporator of step (c); or simultaneously in more than one of the places indicated above.

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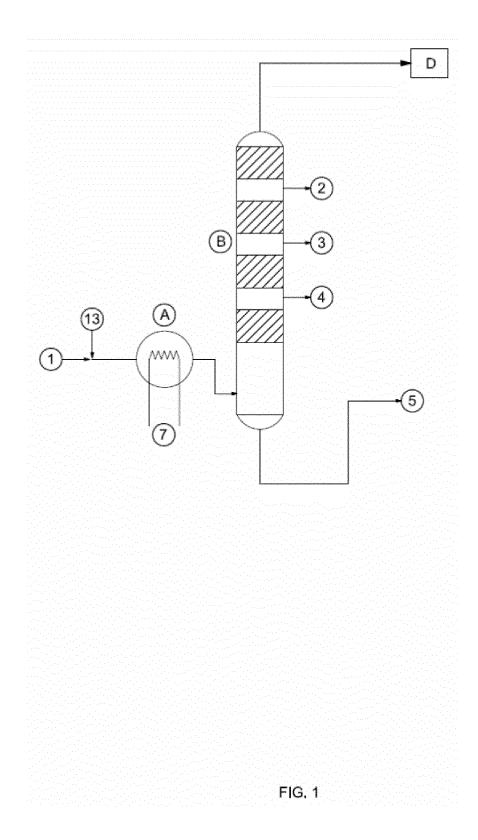
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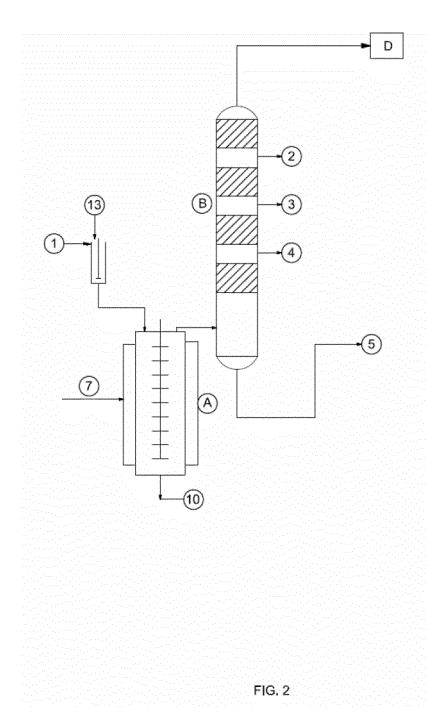
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- 9. The process according to anyone of previous claims, wherein the viscosity of the bottom product obtained in step (c) is reduced by mixing it with a fluxing fluid.
- **10.** The process according to claim 9, wherein the fluxing fluid is a part or the total of the light fraction obtained in step (b) as first side cut of the fractionation column.
 - **11.** The process according to claim 9, wherein the fluxing fluid is the heavy gas-oil contained in the light base oil as first side cut obtained in step (b), that is obtained by increasing the temperature at the top of the fractionation column and condensing the vapors that distill over.
 - **12.** The process according to anyone of previous claims, wherein the distillation of step (b), or the evaporation of step (c) or both are carried out in the presence of a basic compound.
- 13. The process according to claim 12, wherein the basic compound is an alkaline hydroxide or a mixture of alkaline hydroxides, preferably in an amount below 12 kg per metric ton of base oil fraction fed to the distillation step (b).
 - 14. The process according to claim 1, wherein the separation of step (a) is carried out by solvent extraction.
- **15.** The process according to claim 14, wherein the separation of step (a) is carried out by solvent extraction with liquid propane.
 - 16. The process according to claim 1, wherein the separation of step (a) is carried out by chemical demetallization.
- 17. The process according to claim 1, wherein the separation of step (a) is carried out by thin film vacuum evaporation.

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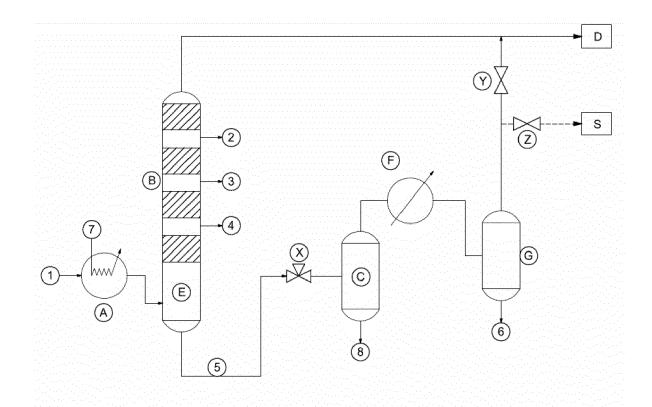


FIG. 3

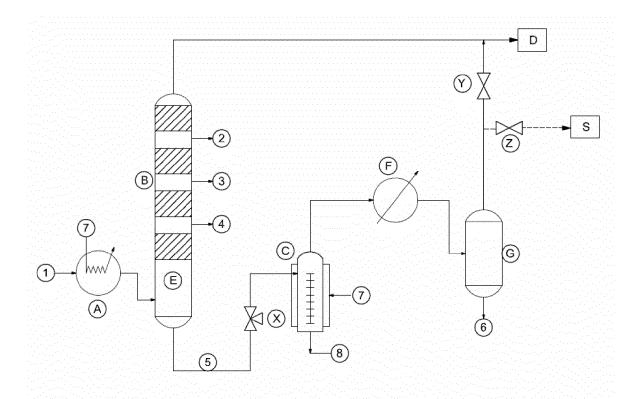


FIG. 4

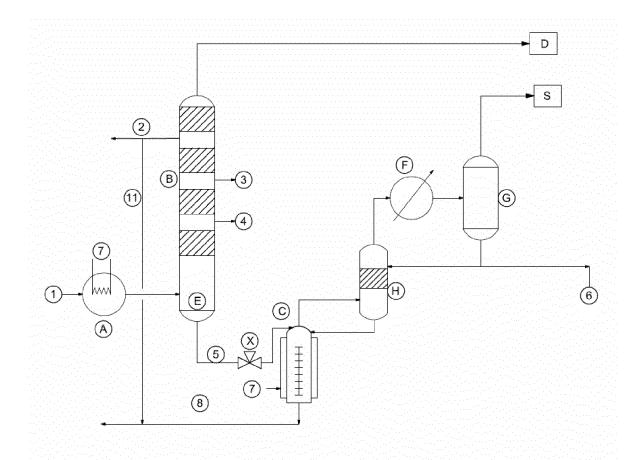
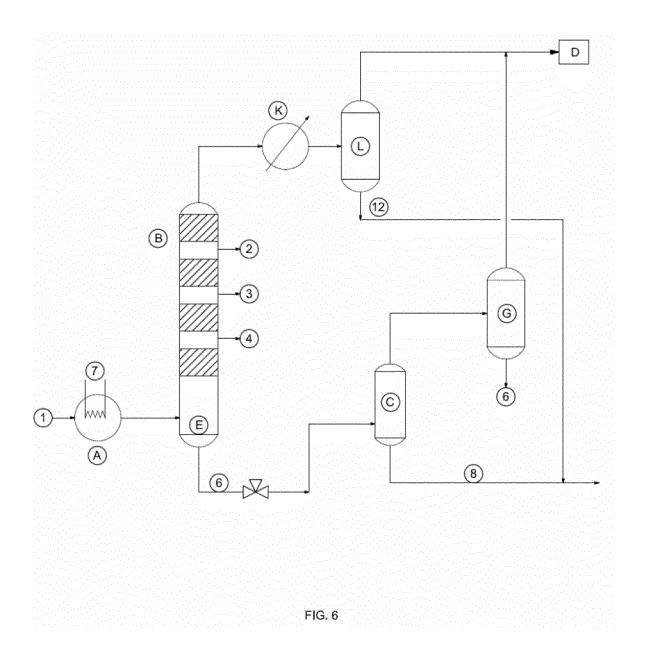


FIG. 5



INTERNATIONAL SEARCH REPORT

International application No. PCT/ES2013/070770

5						
		ssification and IPC				
40	B. FIELDS SEARCHED					
10	Minimum doo	cumentation searched (classification system followed by classi	fication symbols)			
	Documentation	nat such documents are includ	ed in the fields searched			
15	Electronic da	ta base consulted during the international search (name of data	base and, where practicable, s	search terms used)		
	EPODOC, INVENES, WPI, TXTUS, TXTEP, TXTGB					
	C. DOCUMENTS CONSIDERED TO BE RELEVANT					
20	Category*	Citation of document, with indication, where appropriate,	of the relevant passages	Relevant to claim No.		
	A	ES 2169748T T3 (INST FRANCAIS DU PETRO pág. 3, lines 50-62; pág. 4, lines 1-17	LE) 16/07/2002,	1-17		
25	A	WO 2010050901 A1 (ANNDA ENERGY PTE LT AL.) 06/05/2010, paragraphs [0010]-[0015]; fig. 1-2	1-17			
30	A	US 6712954 B1 (POEHLER JOACHIM ET AL. column 2, lines 16-50; column 3, lines 32-50) 30/03/2004,	1-17		
35						
40	* Special	categories of cited documents: "T"		ter the international filing date or		
45	"A" document defining the general state of the art which is not considered to be of particular relevance. "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure use, exhibition, or other means. "P" document published prior to the international filing date but priority date and not in conflict with the application but cit to understand the principle or theory underlying to invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered involve an inventive step when the document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other document.			levance; the claimed invention el or cannot be considered to nen the document is taken alone levance; the claimed invention tolve an inventive step when the		
50	later the	an the priority date claimed "&" ctual completion of the international search	such combination being obv document member of the san Date of mailing of the intern	rious to a person skilled in the art me patent family ational search report		
	OFICINA ES	uling address of the ISA/ PAÑOLA DE PATENTES Y MARCAS	(17/06/ Authorized officer I. González Balseyro	(2014)		
55		astellana, 75 - 28071 Madrid (España) .: 91 349 53 04	Telephone No. 91 3496881			
	Form PCT/IS	A/210 (second sheet) (July 2009)				

	INTERNATIONAL SEARCH	REPORT	International application N	0.
r	Information on patent family members		PCT/ES2013/070770	,
5	Patent document cited in the search report	Publication date	Patent family member(s)	Publication date
10	ES2169748T T3	16.07.2002	KR100372802B B1 US5843384 A US5759385 A JPH08199185 A JP4051488B B2	26.04.2003 01.12.1998 02.06.1998 06.08.1996 27.02.2008 14.08.1996
15			CN1128789 A CN1100854C C PL310964 A1 PL177602B B1 EP0708174 A1 EP0708174 B1	05.02.2003 29.04.1996 31.12.1999 24.04.1996 12.12.2001
20			DE69524533T T2 CA2160652 A1 CA2160652 C EG20615 A NO954097 A NO313296B B1 FR2725725 A1 FR2725725 B1	29.05.2002 18.04.1996 09.10.2007 30.09.1999 18.04.1996 09.09.2002 19.04.1996 13.12.1996
25	WO2010050901 A1	06.05.2010	US2011259795 A1 AU2009310445 A1 SG161129 A1	27.10.2011 06.05.2010 27.05.2010
30	US6712954 B1	30.03.2004	ZA200103751 A UA69426 C2 SK6362001 A3 SK285213B B6 RU2217484 C2	19.12.2001 17.09.2001 06.11.2001 07.09.2006 27.11.2003
35			PT1141181E E PL348757 A1 PL191398B B1 JP2002529579 A JP4246397B B2 HU0104072 A2	30.06.2004 03.06.2002 31.05.2006 10.09.2002 02.04.2009 29.05.2002
40			HU0104072 A3 ES2222051T T3 WO0027957 A1 EP1141181 A1 EP1141181 B1 DK1141181T T3	30.08.2004 16.01.2005 18.05.2000 10.10.2001 11.02.2004 14.06.2004
45			CZ20011559 A3 CZ298571 B6 CN1326498 A CN1185327C C CA2351606 A1 CA2351606 C	12.12.2001 07.11.2007 12.12.2001 19.01.2005 18.05.2000 07.06.2005
50			BR9916606 A BR9916606 B1 AU1161900 A	14.08.2001 18.10.2011 29.05.2000
55	Form PCT/ISA/210 (patent family annex) (July 2009)			

	INTERNATIONAL SEARCH REPORT		International application No.		
	Information on patent family members			PCT/ES2013/070770	
5	Patent document cited in the search report	Publication date	Patent family member(s)	Publication date	
10			AT259405T T DE19852007 A1 DE19852007 C2	15.02.2004 18.05.2000 13.06.2002	
15					
20					
25					
30					
35					
40					
45					
50					
55	Form PCT/ISA/210 (patent family annex) (July 2009)				

Form PCT/ISA/210 (patent family annex) (July 2009)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- WO 9407798 A **[0005]**
- NL 8304023 [0006]
- EP 574272 A [0006]
- WO 9421761 A [0007]
- BE 873451 **[0007]**

- ES 2199697 A1 [0007]
- US 7226553 B2 [0009]
- WO 9826031 A1 [0009]
- US 7226533 B2 [0056]