

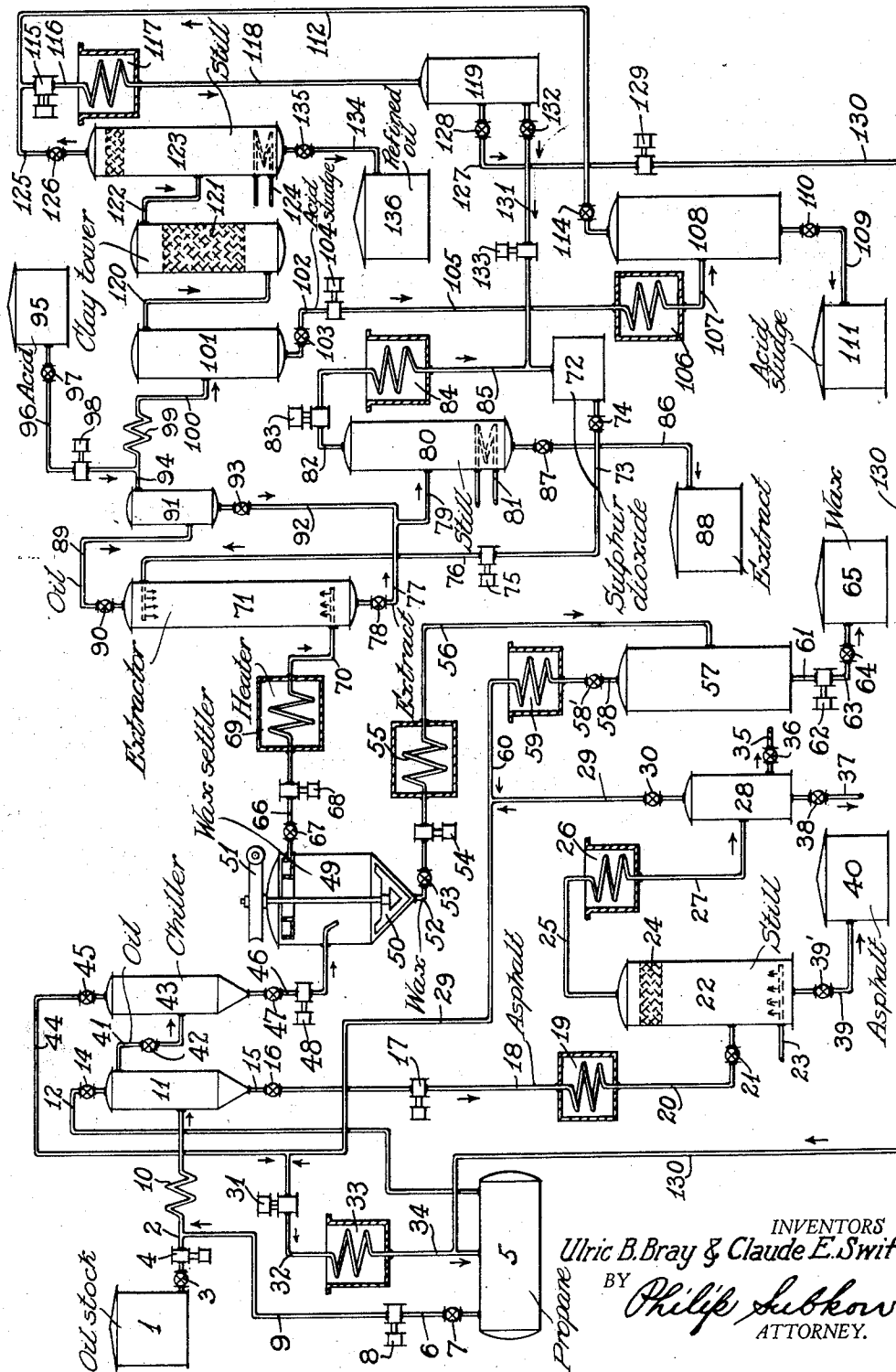
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PROCESS FOR THE PRODUCTION OF LUBRICATING OIL

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PROCESS FOR THE PRODUCTION OF
LUBRICATING OIL

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This invention relates to a process for treating petroleum or fractions thereof to produce lubricating oil and is a continuation in part of applications Serial Nos. 555,017 and 555,018, filed August 4, 1931.

One of the distinctive characteristics of a lubricating oil is its viscosity. For many purposes lubricants are preferred which exhibit a minimum variation in viscosity with variation in temperature, i. e. have low viscosity temperature susceptibilities. It is generally known that the viscosity of lubricating oil produced from western crude oils, such as California naphthene base crudes which are crudes containing asphalt, varies more with change in temperature than lubricating oils produced from eastern crude oils or paraffin base crudes. This means that if two such oils have an identical viscosity at 100° F., the western oil will have a much lower viscosity at the higher temperature of 210° F. than the paraffin base oil. This change in viscosity with temperature is sometimes called the "temperature viscosity susceptibility" of an oil. It is one of the characteristics of western lubricating oils that they comprise distillates, that is, fractions which have been vaporized from crude oil and condensed. Lubricating oils produced from paraffin base oils which contain substantially no asphalt are generally produced by first distilling light oil fractions including gasoline, kerosene and gas oil and also taking overhead light lubricating oils, called "neutrals" having a viscosity in the neighborhood of 100–200 seconds Saybolt universal at 100° F., leaving an undistilled residue termed "bright stock". The lubricating oils of varying viscosity are produced by blending these "neutrals" with the "bright stock" in the desired proportion to produce oil of desired viscosity.

The present invention is related to a process for producing lubricating oils from oils containing asphalt and/or wax. In our process, crude oil containing asphalt and/or wax is topped to remove the lighter fractions of the oil. The topped crude is subsequently treated for removal of the asphalt and/or wax. We prefer to effect the separation of the asphalt from the oil by means of solvents which are capable of dissolving the oil and which do not dissolve the asphalt. Such solvents are light petroleum fractions such as naphtha, casinghead gasoline and petroleum fractions normally vaporous at ordinary temperature and pressure. Other solvents which may be employed are alcohol, ether, mixtures of alcohol and ether, acetone, etc. We prefer

to use as our solvent liquefied normally gaseous hydrocarbons. We intend to include such liquefied normally gaseous hydrocarbons as methane, ethane, propane, butane, isobutane and mixtures thereof. These normally gaseous hydrocarbon solvents may be obtained by rectification of casinghead gasoline by the so-called "stabilizing" method now conventional in the natural gasoline industry. They are the overhead obtained and are liquefied by compression and cooled in the conventional manner and are drawn into pressure chambers where they are maintained in a liquid state until used. A typical analysis of such a fraction is 6.72% ethane, 72.2% propane, 19.91% iso-butane and 1.17% normal butane. Such a fraction may be maintained in a liquid state at a pressure of 125 lbs. per square inch at a temperature of about 75° F.

In carrying out the extraction of the oil with this liquefied normally gaseous hydrocarbon solvent, the solution is maintained at a pressure sufficient to maintain the propane in the liquid state at ordinary temperature of about 70 to 80° F., i. e. at about 125–175 lbs. per square inch. The commingling of the oil with propane at such pressures results in the precipitation of asphalt which is separated from the oil as material substantially free of oil and consisting chiefly of pure bitumen. The remaining oil dissolved in the liquid propane contains substantially all of the lubricating oil components which are present in the crude oil and in substantially the same form as they existed in the original crude oil and also the major portion of the wax present in the crude oil.

The wax present in the oil may be separated by chilling the oil propane mixture to low temperatures to precipitate the wax after which the chilled mass is cold settled, centrifuged or filtered to remove the precipitated wax from the propane oil solution. One method for accomplishing chilling of the oil to precipitate wax is by reducing the pressure on the propane oil mixture subsequent to asphalt separation under high pressure to permit vaporization of propane which lowers the temperature of the oil and causes the wax present to precipitate from solution.

We have discovered that many lubricating oils obtained by the extraction with solvents, such as propane from crude oil containing asphalt and wax, are composed of fractions which have a relatively high gravity viscosity constant, i. e. a relatively high temperature viscosity susceptibility and fractions which have a relatively low gravity viscosity constant, i. e. a relatively low

temperature viscosity susceptibility and that these oils may be separated into oils which exhibit a low temperature viscosity susceptibility resembling oils produced from asphalt free crudes and oils which exhibit a high temperature viscosity susceptibility corresponding to oils produced by distillation of asphaltic crudes.

The gravity viscosity constant has been defined by Hill and Coates in the Journal of Ind. and Eng. Chem., vol. 20, 1928, page 641. This constant represents the paraffinicity or naphthenicity of an oil. A high value represents a high degree of naphthenicity, while a low value indicates a relatively greater paraffinicity. Lubricating oils from natural crudes range from .903 for an extreme Gulf Coast type to .807 for an Eastern Pennsylvania type or even beyond. For convenience, we will call the oils which exhibit a low temperature viscosity susceptibility or gravity viscosity constant as "paraffinoid" or oils of high paraffinicity and those which exhibit a high temperature viscosity susceptibility or gravity viscosity constant and which resemble western lubricating oil distillates as "non-paraffinoid" or oils of low paraffinicity. The propane extract which consists of such a mixture of paraffinoid and non-paraffinoid fractions may be separated into these respective fractions by the use of solvents which will selectively dissolve out the non-paraffinoid components. We have found that certain solvents have a selective solvent action for the non-paraffinoid oils. As solvents which will effect this separation, we have found as useful liquid sulphur dioxide, mixtures of acetone and benzol, aniline, methyl formate, dichlorethyl ether, nitrobenzol, chloraniline, phenol and cresol. Liquid sulphur dioxide, dichlorethyl ether and nitrobenzol have been found especially valuable as solvents to separate the propane extract into oils which exhibit low temperature viscosity susceptibilities and into oils which exhibit high temperature viscosity susceptibilities.

We have also discovered that more efficient separation of the desirable paraffinoid fractions from the undesirable non-paraffinoid fractions and impurities may be effected if the separation or extraction is accomplished in the presence of a liquefied normally gaseous hydrocarbon solvent. We have found it advantageous to carry out the extraction of the oil subsequent to the separation of the asphalt and wax with liquid sulphur dioxide in the presence of the liquid propane used to precipitate and separate the asphalt from the oil. The propane solution of oil is extracted with liquid sulphur dioxide to remove therefrom the non-paraffinoid components. Liquid propane is a good solvent for the paraffinic fractions but a poor solvent for the impurities such as the gums, resins and other compounds which exhibit high carbon forming characteristics. Furthermore, if an oil is extracted under the same conditions of temperature and amounts of selective solvent, i. e. liquid sulphur dioxide, with and without the use of liquid propane, a greater yield of paraffinoid oil is obtained when the extraction is carried out in the presence of the liquid propane. The presence of the liquid propane during the extraction appears to lower the solvent power of the extractive solvent, the result being that paraffinic fractions which are normally dissolved in the extract phase during the extraction without the presence of liquid propane are recovered in the solution of oil and liquid propane in the so-called raffinate phase of extraction.

As a further means of improving the tempera-

ture viscosity susceptibility of the oil, we may treat it with sulphuric acid. Such treatment removes from the oil a portion of the bodies present which possess a high temperature viscosity susceptibility. The treatment with sulphuric acid is preferably followed by treatment with clay to remove entrained sulphuric acid sludge which does not separate by settling and also to remove coloring matter which imparts a poor color to the oil. It is preferable to effect the chemical treatment with sulphuric acid and clay when the oil is dissolved in the liquid propane for the reason that the sludge formed in acid treating the paraffinic fraction or raffinate produced by extraction with liquid sulphur dioxide is a liquid sludge as contrasted to the hard sludge often obtained on treating lubricating stocks directly, that is, without the presence of liquid propane. The soft sludge obtained in acid treating the raffinate becomes emulsified and is difficult to make agglomerate and settle from the oil when the treatment is carried out in the absence of a very low viscosity diluent such as liquid propane.

We have found that by performing the acid treating step in the process subsequent to extraction with liquid sulphur dioxide that more effective results are obtained. By acid treating the oil prior to extraction with liquid sulphur dioxide a large portion of the acid is consumed in removing from the oil unsaturated hydrocarbon bodies which easily are removed with liquid sulphur dioxide. Consequently, by treating the oil with acid subsequent to extraction with liquid sulphur dioxide, the acid utilized in such treatment is free to act upon those undesirable bodies present in the oil which are not removable by liquid sulphur dioxide. Thus, by acid treating the oil subsequent to extraction with liquid sulphur dioxide, we are able to obtain a more economical use of the acid and a more effective removal of the undesirable bodies present in the oil.

It is preferable to perform the wax separation step prior to extraction of the oil with selective solvent into fractions of high paraffinicity and fractions of low paraffinicity for the reason that the low temperatures of extraction necessary with certain selective solvents such as liquid sulphur dioxide, cresol and chloraniline causes the wax to precipitate from solution and this hinders extraction with the selective solvent. For example, the extraction with liquid sulphur dioxide and chloraniline is preferably at 20 to 50° F., and with cresol at -5 to 20° F. depending upon the type of oil treated. For California oils, the preferred treating temperatures are lower than those for mid-continent oils. When treating at temperatures below approximately 35° F., wax, if present, will invariably precipitate from solution and hinder the extraction process. Moreover, it is easier to raise the temperature from a dewaxing temperature to the higher extraction temperature by simple heating than it is to lower the temperature from an extraction temperature to a dewaxing temperature.

With the above discussion in mind, it will be perceived that it is an object of our invention to isolate from crude oil or topped crude oil, those fractions having characteristics which impart to the oil the low temperature viscosity susceptibility typical of paraffin base oil.

More particularly, it is an object of our invention to isolate from asphalt containing oils those components which impart to the oil the reduced susceptibility to change in viscosity with temperature which is characteristic of oils obtained from

non-asphalt containing oils, that is, to separate the paraffinoid from non-paraffinoid components of the asphalt containing crude oils.

It is a further object of our invention to separate asphalt from an oil containing asphalt, oil and wax by means of a solvent, to separate the wax from the oil and solvent and subsequently to separate the asphalt and wax free oil by means of a second solvent used in the presence of the first solvent into a paraffinoid oil and a non-paraffinoid oil. It is a further object to thereafter treat the separated paraffinoid oil dissolved in the first solvent with sulphuric acid.

It is a further object of our invention to employ a normally gaseous hydrocarbon solvent, in particular liquid propane, as the solvent for separating the asphalt from the oil and the use of liquid sulphur dioxide as the solvent for separating the oil into paraffinic and non-paraffinic fractions.

It is a particular object of our invention to separate the oil, subsequent to the removal of asphalt and/or wax from the oil, into paraffinic and non-paraffinic fractions by means of liquid sulphur dioxide employed in the presence of liquid propane and to thereafter treat the paraffinic fraction with sulphuric acid.

Other objects and advantages of our invention will be apparent from the following description of the invention taken from the drawing which refers to a schematic arrangement of apparatus for carrying out the invention.

Referring to the drawing, an asphalt and wax containing oil such as topped residuum from which the lighter fractions, such as gasoline, kerosene and gas oil have been removed, is taken from tank 1 and is drawn into line 2 controlled by valve 3 by pump 4 where it meets a stream of liquefied normally gaseous hydrocarbon solvent such as liquid propane taken from storage tank 5 via line 6, controlled by valve 7 and pump 8 which forces the liquid propane via line 9 into line 2. The amount of propane introduced into line 2 will depend upon the character of the oil and the temperatures desired in the wax precipitating decanter or chilling column 43. The mixture of liquid propane and oil containing asphalt and wax is passed through turbulence or mixing coil 10 into asphalt precipitator or decanter 11 where the asphalt present in the oil is precipitated and settled rapidly to the bottom of the decanter as a slurry of substantially pure bitumen or hard asphalt containing some oil and propane. A pressure of approximately 25 to 175 lbs. per square inch and a temperature of about 70 to 80° F. is maintained in the decanter 11. The pressure in decanter 11 is maintained by means of equilibrium line 12 controlled by valve 14 which is connected to propane storage tank 5.

The amount of propane introduced into the oil will also depend upon whether it is desired to remove the asphalt at the bottom of the decanter 11 as a slurry of asphalt and propane, i. e. as a mixture of substantially pure bitumen or hard asphalt and propane or as a liquid fraction of asphalt containing propane and some oil. If it is desired to separate the asphalt in decanter 11 as a liquid fraction, the volumetric ratio of propane to oil in decanter 11 will be within the range of 0.6 to 6 to 1, depending upon the character of the oil and the degree to which it was topped. Higher volumetric ratios will precipitate and settle the asphalt as a slurry of substantially pure bitumen and propane containing very little oil.

The asphalt precipitated in decanter 11 is withdrawn via line 15 controlled by valve 16 and

is forced by pump 17 under pressure through line 18 and heating coil 19 where it is heated to a sufficiently high temperature to melt the asphalt and vaporize residual propane. The heated mixture is then passed through line 20 and flashed through pressure reduction valve 21 into evaporator 22 which operates at a lower pressure. Superheated steam is introduced into the evaporator through perforated pipe 23 to supply additional heat and to reduce the asphalt to proper specification generally only, as regards flash and fire points. The overhead from evaporator 22 passing through mist extractor 24 is sent through line 25 to cooler 26 and then via line 27 into separator 28. The uncondensed propane from separator 28 is sent through line 29 controlled by valve 30 to the suction of compressor 31 where its pressure is raised to that in the high pressure system and is then sent via line 32 to cooler 33 where it liquefies and runs down into propane storage tank 5 via line 34. Any condensed light oil in separator 28 is withdrawn via line 35 controlled by valve 36 and condensed water through line 37 controlled by valve 38. The asphalt is taken from the bottom of evaporator 22 via line 39 controlled by valve 39' and passed into storage tank 40.

The overflow from decanter 11 consisting of propane and oil free from asphalt but containing wax is sent through line 41 and pressure reduction valve 42 into the wax precipitating decanter or chilling column 43. In chiller 43, sufficient propane vaporizes to reduce the temperature of the remaining oil and propane to a sufficiently low temperature which causes wax to precipitate from solution. It is preferable to gradually lower the temperature in the chiller 43 at a rate not in excess of 4° F. which is accomplished by controlling the pressure in the chiller 43 by the proper operation of valve 45 on line 44 and compressor 31. The pressure will be gradually lowered in chiller 43 to about 0 to 25 lbs. gauge which corresponds to a temperature of approximately -40° F. to 0° F. If desired, the temperature may be lowered rapidly, as for example, at a rate of approximately 10° F. per minute until a temperature of approximately 35 to 45° F. is reached after which the temperature may be lowered gradually to approximately -40° F. to 0° F. at a rate of about 3° F. per minute. If sufficient propane is not present in the oil passing into the chiller 43 to obtain the desired low temperature for wax precipitation, additional propane may be introduced into the oil passing into chilling column 43 to provide for the desired low temperature upon subsequent vaporization of propane. The propane evaporating in the chiller 43 will pass out of the top through line 44 controlled by valve 45, to the suction of compressor 31 where the vapors are compressed which are then cooled in cooler 33 and run down into propane storage tank 5.

Due to the fact that sufficient time must be given to gradually chill the solution of oil, wax and propane passing into the chiller 43 and to make the process continuous, a plurality of chilling columns may be provided which may be operated alternately. However, when operating a batch process merely one of such chilling columns will be sufficient. Thus, the entire charge from decanter 11 may be transferred to the column 43 and the chilling may be accomplished by controlling the operation of valve 45 on line 44.

The solution of propane and oil containing the wax precipitated by internal refrigeration is

withdrawn from chiller 43 via line 46 controlled by valve 47 and pumped by pump 48 into the vapor tight separator or settler 49 where sufficient time is provided for allowing the precipitated wax to settle from solution to the bottom of the settler. In batch operation only one of these settlers will suffice but to make the process continuous a plurality of such settlers may be provided which may be operated alternatively in order to provide for sufficient settling periods. In order to prevent ebullition in the wax separator during the wax settling period, pressure is imposed upon the solution of oil. This is accomplished by maintaining pressure within the separator by pump 48. As the chilled mass in the wax separator remains in a nonebullient state, the wax settles out and is collected by vanes 50 operated by pulley 51 connected to a suitable source of power not shown. If sufficient propane is not present in the wax settler 49 to give good settling of the precipitated wax, additional propane may be introduced into the wax settler from the propane storage tank 5. Generally, a volumetric ratio of substantially not less than four volumes of propane to one of oil should be maintained during the wax settling operation. The precipitated wax settling at the bottom of the wax separator 49 as a slurry of wax and propane is removed via line 52 controlled by valve 53 and pumped by pump 54 through heater 55 and through line 56 into separator 57. Vaporized propane is passed to the storage tank 5 via line 58 controlled by valve 58', cooler 59, lines 60 and 29, compressor 31, cooler 33 and line 34. The propane free wax is withdrawn from the separator 57 through line 61 and pumped by pump 62 into line 63 controlled by valve 64 into storage tank 65.

The solution of oil in liquid propane and free from asphalt and wax is withdrawn from the wax settler 49 via line 66 controlled by valve 67 and pumped by pump 68 through heater 69 where its temperature is raised to an extraction temperature with liquid sulphur dioxide of approximately 20 to 50° F. and even higher. The propane solution of oil is then passed via line 70 into the lower zone of extraction column 71. Liquid sulphur dioxide from tank 72 is withdrawn via line 73 controlled by valve 74 and pumped by pump 75 through line 76 into the upper zone of extraction column 71. Due to the difference in specific gravity of the oil introduced into the lower zone of the extraction column and the liquid sulphur dioxide introduced into the upper zone of the extraction column, these two liquids tend to separate. As the liquid sulphur dioxide descends through the extraction column, it dissolves certain components present in the oil comprising the fractions in the oil of low paraffinicity leaving undissolved the fractions of high paraffinicity dissolved in the liquid propane. It is preferable to effect the extraction with liquid sulphur dioxide in the presence of two volumes of propane to one volume of oil. Approximately two to three volumes of liquid sulphur dioxide should be employed to obtain the desired separation of the oil into paraffinic and non-paraffinic fractions.

The solution of liquid sulphur dioxide and oil is removed from the extraction column 71 via line 77 controlled by valve 78 and sent by means of line 79 into vaporizer 80 where the sulphur dioxide present in the oil is vaporized by aid of steam circulated through closed coil 81 and passes through line 82 to compressor 83 and then to condenser 84 where the sulphur dioxide is liquefied and sent to storage tank 72 via line 85. The

sulphur dioxide soluble constituents of the oil or extract is removed from the bottom of the vaporizer 80 via line 86 controlled by valve 87 and sent to storage tank 88.

The ascending column of oil in the extraction column 71 dissolved in liquid propane and from which the liquid sulphur dioxide soluble components have been removed passes through valve 90 on line 89 into an auxiliary separator 91 where any remaining liquid sulphur dioxide present in the oil and components soluble in liquid sulphur dioxide which were not settled in extraction column 96 are settled out and pass via line 92, controlled by valve 93 into line 79 to the vaporizer 80.

The clear solution of oil and propane is decanted from the auxiliary separator 91 and passes into line 94 where it meets a stream of sulphuric acid or other acids coming from storage tank 95 via line 96 controlled by valve 97 and pump 98. The mixture of acid, oil and propane passes from line 94 through mixing or agitating coil 99 where the mixture is thoroughly agitated after which it passes through line 100 into the separator 101 where the sulphuric acid sludge is allowed to settle out. If sufficient propane is not present in the oil to permit good settling of the acid sludge, further quantities may be introduced into the oil into line 94 prior to the sulphuric acid treatment or it may be added into line 100 subsequent to the agitation with sulphuric acid. The acid sludge settling to the bottom of separator 101 is withdrawn via line 102 controlled by valve 103 and pumped by pump 104 through line 105 through heater 106 where the mass is heated sufficiently to vaporize propane present in the sludge. The heated mass passes from heater 106 through line 107 into separator 108 where the sludge separates from the propane and is withdrawn via line 109 controlled by valve 110 and sent to storage 111. The vaporized propane is drawn into line 112, controlled by valve 114, compressed in compressor 115 and passed via line 116 to condenser 117 where it is liquefied and sent by means of line 118 to separator 119 where it is separated from any liquid sulphur dioxide.

The acid treated oil in separator 101 dissolved in propane and carrying varying amounts of liquid sulphur dioxide is withdrawn via line 120 and passed into clay tower 121 where the oil and propane is treated with fuller's earth, Death Valley 50 clay, diatomaceous earth, bentonite, charcoal or the like for the purpose of removing any remaining sludge and neutralizing the oil. The fraction of oil leaving clay tower 121 passes by means of line 122 to evaporator 123 where the propane and sulphur dioxide present in the oil are vaporized by the aid of steam circulated through closed coil 124 and are sent via line 125 controlled by valve 126 to compressor 115, condenser 117, via line 118 into separator 119 where the propane is separated from the liquid sulphur dioxide and passes via line 127 controlled by valve 128 and pumped by pump 129 via line 130 to propane storage tank 5. The liquid sulphur dioxide in separator 119 is withdrawn via line 131 controlled by valve 132 and pumped by pump 133 to liquid sulphur dioxide storage tank 72. The treated oil in evaporator 123 which has been freed from propane and sulphur dioxide is removed via line 134 controlled by valve 135 and passed to refined oil tank 136.

The foregoing example is merely illustrative of a preferred mode of carrying out our invention and is not to be taken as limiting as many vari-

ations may be made within the scope of the following claims by any one skilled in the art:

We claim:

1. A process for the treatment of hydrocarbon oil containing wax which comprises commingling said oil with a liquefied normally gaseous hydrocarbon diluent, chilling said oil and diluent to precipitate wax, separating the wax from the oil and diluent, extracting the dewaxed and diluted oil with a selective solvent for non-paraffinoid fractions to form an oil fraction dissolved in said diluent and an oil fraction dissolved in said solvent and separating said fractions.
2. A process according to claim 1 in which the diluent is liquid propane.
3. A process for the treatment of hydrocarbon oil containing wax which comprises dissolving said oil under superatmospheric pressure in a liquefied normally gaseous hydrocarbon diluent, releasing the pressure and vaporizing a portion of said diluent to chill the oil and precipitate wax, removing the wax from the oil dissolved in the remaining diluent, extracting the oil dissolved in said remaining diluent with a selective solvent for non-paraffinoid fractions to form an oil fraction dissolved in said diluent and an oil fraction dissolved in said solvent and separating said fractions.
4. A process according to claim 1 in which the selective solvent is liquid sulphur dioxide.
5. A process according to claim 1 in which the oil fraction dissolved in the diluent is treated with acid.
6. A process according to claim 1 in which the oil fraction dissolved in the diluent is treated with acid and clay.
7. A process for the treatment of oil containing asphalt and wax which comprises commingling said oil with an asphalt precipitating solvent to precipitate asphalt, removing the asphalt from the waxy oil dissolved in said solvent, chilling the solvent solution of waxy oil to precipitate wax, removing the wax from the oil and solvent, extracting the dewaxed and deasphaltized oil dissolved in said asphalt precipitating solvent with a selective solvent for non-par-

affinoid fractions to form a fraction dissolved in said selective solvent and a fraction dissolved in said asphalt precipitating solvent and separating said fractions.

8. A process according to claim 7 in which the selective solvent is liquid sulphur dioxide.

9. A process according to claim 7 in which the asphalt precipitating solvent is a liquefied normally gaseous hydrocarbon.

10. A process according to claim 3 in which the liquefied normally gaseous hydrocarbon is liquid propane.

11. A process for the treatment of oil containing asphalt and wax which comprises commingling said oil with a liquefied normally gaseous hydrocarbon diluent to precipitate asphalt, removing the precipitated asphalt from the waxy oil dissolved in said diluent, vaporizing a portion of said diluent to chill the oil and precipitate wax, removing the precipitated wax from the oil and remaining diluent, extracting the dewaxed and deasphaltized oil dissolved in the remaining diluent with a selective solvent for non-paraffinoid fractions to form a fraction dissolved in said diluent and a fraction dissolved in said solvent and separating said fractions.

12. A process according to claim 3 in which the selective solvent is liquid sulphur dioxide.

13. A process for the treatment of hydrocarbon oil containing wax which comprises commingling said oil with a liquefied normally gaseous hydrocarbon diluent under superatmospheric pressure, chilling the diluted oil to precipitate wax, separating the precipitated wax from the diluted oil, extracting the dewaxed and diluted oil with a selective solvent for the non-paraffinoid fractions to form an oil fraction dissolved in said diluent and an oil fraction dissolved in said solvent, separating the dewaxed and extracted oil dissolved in said diluent from the oil dissolved in said solvent and treating the dewaxed and extracted oil dissolved in said diluent with sulphuric acid.

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