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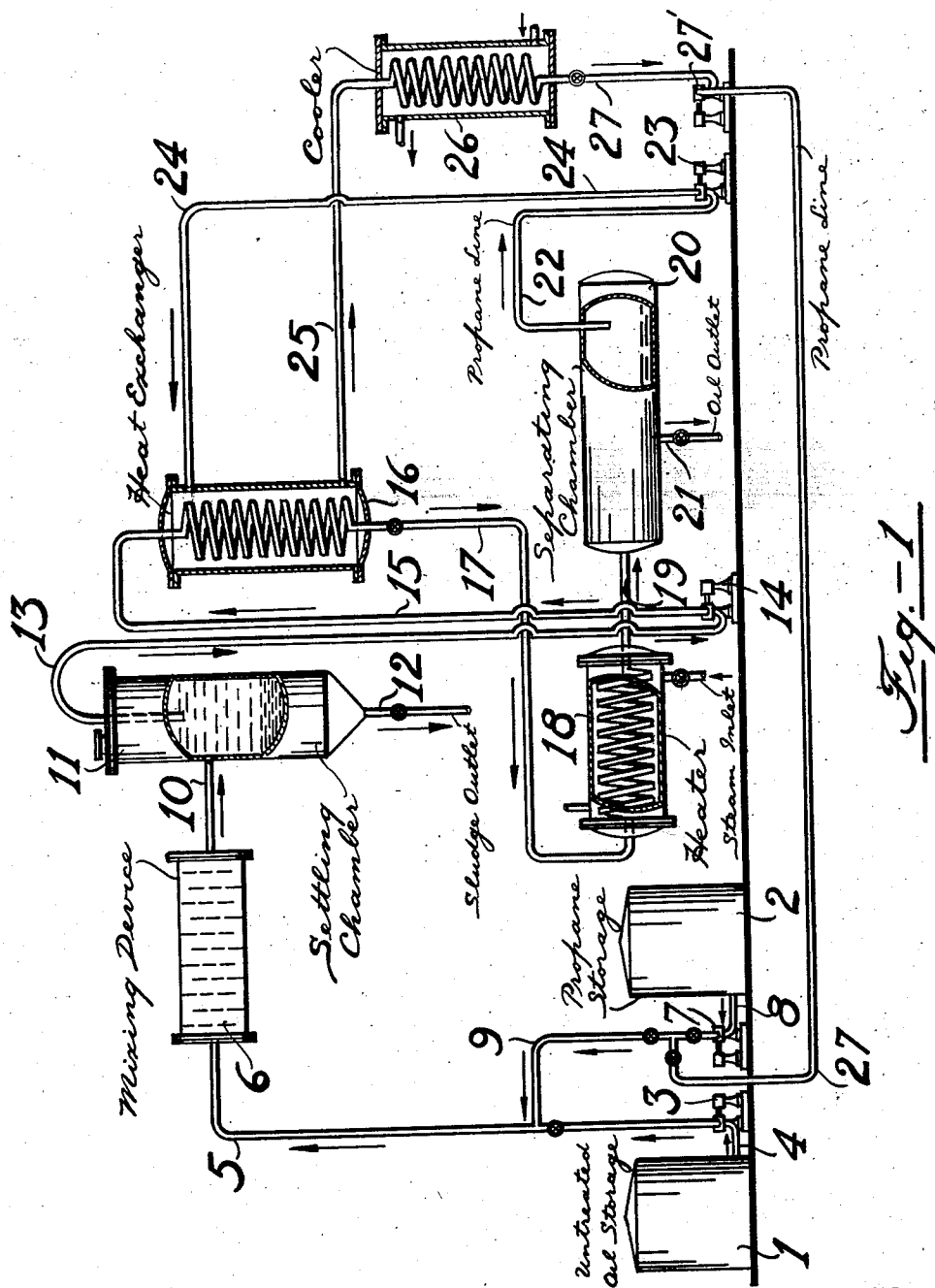
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2,115,003

RECOVERY OF SOLVENT IN SOLVENT TREATING PROCESSES

Filed July 30, 1932

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

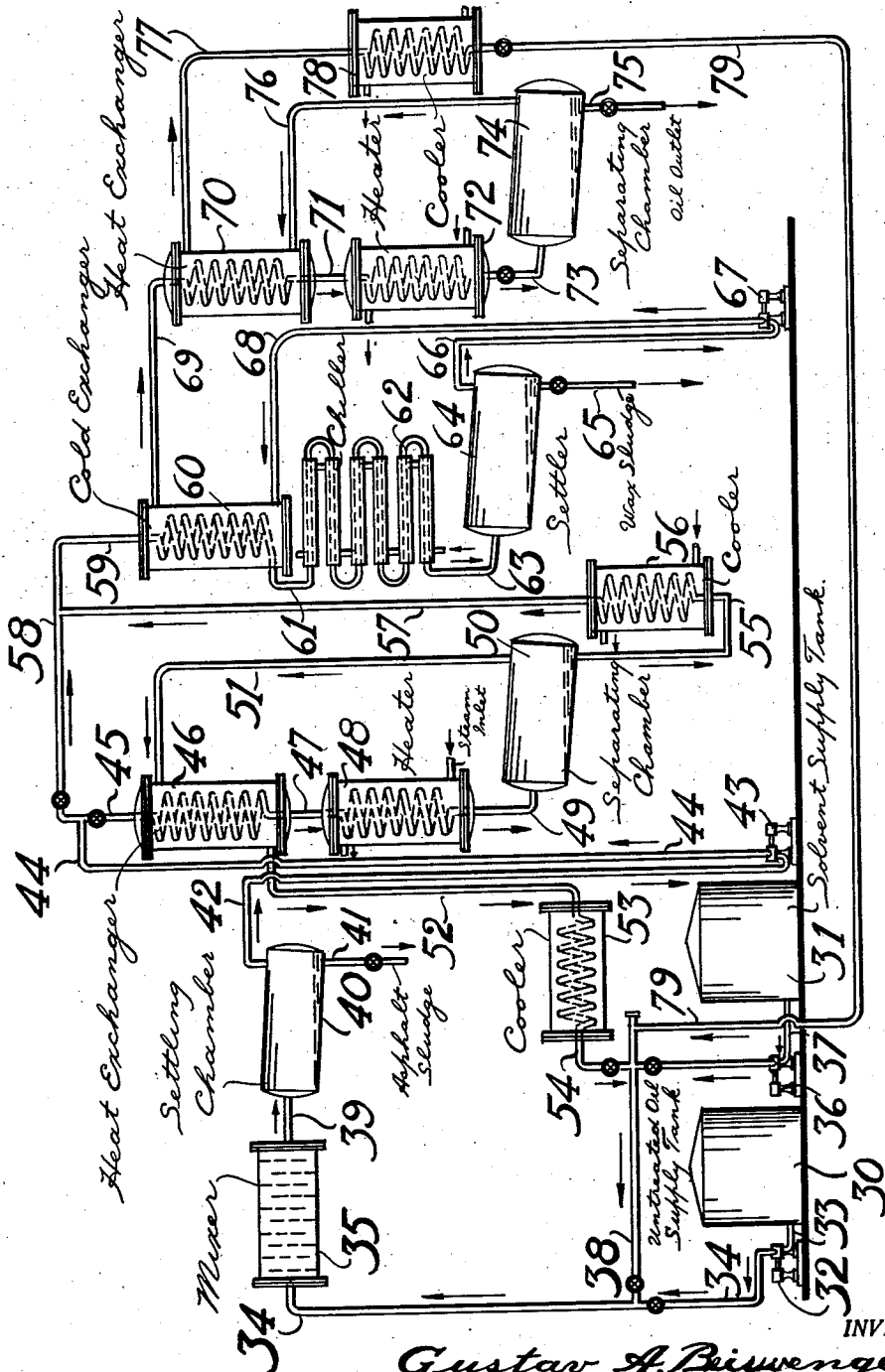


Fig. - 2

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UNITED STATES PATENT OFFICE

2,115,003

RECOVERY OF SOLVENT IN SOLVENT
TREATING PROCESSESGustav A. Beiswenger, Elizabeth, N. J., assignor
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14 Claims. (Cl. 196—13)

This invention relates to the treatment of hydrocarbon oils with a selective solvent and more specifically relates to the recovery of the solvent from the treated oil. The invention has particular reference to the treatment of heavy hydrocarbon oils with light hydrocarbons.

The term "heavy hydrocarbon oil" will be understood to mean lubricating distillates, cylinder oils, topped crudes, crude bottoms and tarry or asphaltic residues or sludges, whether obtained by the distillation, cracking or destructive hydrogenation of petroleum oils, shale oils, coals, tars, pitches, bitumens or other carbonaceous material, or whether prepared synthetically by the polymerization of hydrocarbons in the presence of agents such as aluminum chloride or by the condensation of aromatic and paraffinic hydrocarbons in the presence of condensing agents such as for example the condensation products of halogenated paraffin wax and naphthalene.

The term "light hydrocarbons" will be understood to mean liquefied hydrocarbons or mixtures of liquefied hydrocarbons of one to five carbon atoms, such as for example, ethane, propane, butane, pentane, or mixtures of these.

In the treatment of heavy hydrocarbon oils with light hydrocarbons it is necessary to remove the light hydrocarbon solvent from the treated oil. This may be accomplished by simple distillation. I have found, however, that it may be more advantageously accomplished by heating the oil-solvent mixture to a temperature at which the oil becomes substantially insoluble in the solvent, and then separating the oil and solvent layers.

My invention therefore comprises heating a mixture of hydrocarbon oil and light hydrocarbon solvent to a temperature close to the critical temperature of the light hydrocarbon, if the solvent comprises a single light hydrocarbon, or to a temperature between the critical temperatures of the two light hydrocarbon components if the solvent comprises two light hydrocarbons, whereby the oil becomes substantially insoluble in the solvent and then separating the two layers.

Two important advantages of separating the solvent from the heavy hydrocarbon oil in this manner over separation by true distillation are that distillation and condensation equipment may be largely dispensed with, and heat exchange and other operations are greatly simplified.

The method of carrying out the separation will be fully understood from the following de-

scription read with reference to the accompanying drawings which are semi-diagrammatic views in sectional elevation of suitable types of apparatus for my purposes and of which

Figure 1 illustrates the simplest embodiment of the invention in which the oil is treated with solvent in a single stage and the solvent is then recovered from the treated oil and returned to the treating step, and

Figure 2 illustrates another embodiment of the invention in which the solvent recovery method is applied to a process for deasphalting and dewaxing a heavy hydrocarbon oil in the presence of light hydrocarbons.

Referring to Figure 1, numeral 1 designates a supply tank for the oil to be treated and numeral 2 denotes a supply tank for the light hydrocarbon solvent which for convenience in description may be referred to as propane. Pump 3 draws oil from tank 1 through line 4 and forces it through line 5 into a mixing device 6 which may be of any suitable type. Pump 7 draws propane from tank 2 through line 8 and forces it through lines 9 and 5 also into mixing device 6, wherein the oil and solvent are thoroughly commingled. From mixer 6 the flux of oil and solvent flows through line 10 into a settling chamber 11 wherein normally solid materials contained in the oil, such as asphaltic and resinous bodies, which are thrown out by the propane, are allowed to settle. The sludge of asphaltic or resinous material containing some propane is drawn off from settler 11 through line 12. The small amount of propane present in this sludge can be removed by distillation (equipment not shown). The remainder of the material in settler 11 which comprises the bulk of the oil and solvent is removed therefrom through line 13 by pump 14 and forced through line 15 into and through a heat exchanger 16 and thence through line 17 into a heater 18 wherein the oil-solvent mixture is heated to a temperature close to the critical temperature of the solvent. In this case, in which the solvent comprises propane, the mixture will be heated to a temperature between, say, 180 and 204° F. (the approximate critical temperature of propane). The hot flux then flows through line 19 into a separating chamber 20 wherein the oil being substantially insoluble in the propane at this temperature settles to the bottom. The oil which contains some propane is withdrawn therefrom through line 21 and its propane content removed by distillation (equipment not shown). The propane is removed through line 22 and forced

by pump 23 through line 24 into and through heat exchanger 16. It flows thence through line 25 into a cooler 26 from which it is returned by means of pump 27 through lines 27, 9 and 5 to the mixer 8 where it may be used to treat fresh oil.

Referring to Figure 2 which illustrates a type of apparatus adapted for deasphalting and dewaxing a hydrocarbon oil and for recovering the solvent in accordance with the herein described method, numeral 30 designates a supply tank for oil to be treated and numeral 31 designates a supply tank for the solvent which again for convenience in description may be referred to as propane. Pump 32 draws oil from tank 30 through line 33 and forces it through line 34 into mixer 35. Pump 36 draws propane from tank 31 through line 37 and forces it through lines 38 and 34 also into mixer 35. The flux of oil and propane flows through line 39 into a settling chamber 40 wherein precipitated asphaltic material is allowed to settle. This is removed through line 41 and its propane content removed by distillation (equipment not shown). The remaining solution in settler 40 is withdrawn therefrom through line 42 by pump 43. Since the solvent concentration in this solution is considerably higher than that desirable for dewaxing, part of the solution is passed through line 45 into and through a heat exchanger 46, whence it leaves through line 47 and is passed into a heater 48 wherein it is heated to a temperature between say 180 and 204° F. (the approximate critical temperature of propane). The heated material then flows through line 49 into a separating chamber 50 wherein the oil, which is substantially insoluble in the propane at this temperature, settles to the bottom. The upper layer, which comprises substantially oil-free propane, passes out through line 51 into and through heat exchanger 46, partially preheating the cold solution, thence through line 52, cooler 53 and line 54 into line 38 to be reused in the process. The oil layer is withdrawn from settler 50 through line 55 and passed through cooler 56. The cooled oil passes through line 57 and mixes with by-passed solution coming through line 58. By controlling the proportional quantities of solution passed through lines 45 and 58 the concentration of oil in the solution resulting from the mixing of the streams from lines 57 and 58 can be maintained at any value which is found to be desirable for the dewaxing step. Thus for example it is generally desirable to use about 10 volumes of solvent per volume of oil in the deasphalting operation whereas about 4 volumes of solvent per volume of oil is a suitable proportion for dewaxing. The mixed solution passes through line 59 into and through a cold exchanger 60, whence it leaves through line 61 and passes through a chilling means 62 wherein it is chilled to a temperature below the wax separation point of the oil. Chilling means 62 may be of any suitable type such as the double pipe type illustrated, and any suitable refrigerant may be used. The chilled flux thence flows through line 63 into a settler 64 wherein the wax is allowed to settle out. The wax sludge is withdrawn through line 65. The remaining dewaxed flux is withdrawn from settler 65 through line 66 by pump 67 and forced through line 68 into and through the cold exchanger 60 wherein it serves partially to chill the entering wax-containing flux. From the cold exchanger 60 the partially warmed dewaxed solution flows through line 69 into and through a heat exchanger 70 and thence

flows through line 71 into a heating means 72 wherein it is heated to a temperature between say 180 and 204° F. (the approximate critical temperature of propane). The heated material then flows through line 73 into a separating chamber 74 wherein the oil which is substantially insoluble in the propane at this temperature settles to the bottom. The oil layer which contains some propane is withdrawn through line 75 and its propane content may be removed by distillation (equipment not shown). The solvent which is substantially free from oil is removed through line 76 and passed into and through heat exchanger 70 wherein it serves partially to preheat cold dewaxed flux withdrawn from the chiller. From heat exchanger 70 the partially cooled recovered solvent flows through line 77 into cooler 78 and thence may be returned by lines 79 and 38 to the mixer 35 where it may be used to treat fresh oil.

In carrying out my method of solvent recovery the treated flux of oil and solvent should in general be heated to a temperature within 30° F. below and 30° F. above the critical temperature of the solvent, preferably within 10° F. above or below the critical temperature. This is true, however, only if the solvent comprises a single light hydrocarbon. If a mixed solvent comprising two light hydrocarbons is used, the flux should be heated to a temperature between the respective critical temperatures of the two light hydrocarbons. The exact temperature to which it must be heated will, of course, depend upon the particular light hydrocarbons used in the solvent and the proportions in which they are present therein.

The pressure maintained on the flux of oil and solvent while being heated to temperatures close to the critical should preferably be within 50 pounds per square inch above or below the vapor pressure of the pure solvent at the particular temperature to which the flux is heated.

When the flux is heated to below the critical temperature and maintained above the vapor pressure of the solvent, the solvent is substantially in the liquid state. When however the flux is maintained below the critical temperature and below the vapor pressure of the solvent, the solvent may be considered to be in the vapor state. At temperature above the critical the solvent may be in the homogeneous phase, which is neither liquid nor vapor. I have found that when operating within 20° F. of the critical temperature and within 50 pounds per square inch of the vapor pressure of the solvent, the variation in physical characteristics of the solvent in either the liquid, vapor or homogeneous phase is so small that the process can be operated successfully within these limits regardless of the state in which the solvent may exist.

This method of recovering a light hydrocarbon solvent from oil-light hydrocarbon mixtures is applicable to all types of processes in which a heavy hydrocarbon material is treated with a light hydrocarbon solvent. It will be understood that various modifications of the method may be made to adapt it to use in any particular combination of treating steps.

This invention is not limited by any theory of its mechanism nor by any details which have been given merely for purpose of illustration, but is limited only in and by the following claims in which I wish to claim all novelty inherent in the invention.

I claim:

1. In a process for treating heavy hydrocarbon oils with a light hydrocarbon solvent the method of recovering the solvent from the treated oil which comprises heating the oil solvent mixture to a temperature and pressure near the critical temperature and pressure of the solvent; and separating the oil layer from the solvent layer.
2. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 30° F. below and 30° F. above the critical temperature of the solvent.
3. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 20° F. below and 20° F. above the critical temperature of the solvent.
4. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 10° F. below and 10° F. above the critical temperature of the solvent.
5. Process according to claim 1 in which the oil solvent mixture is heated under a pressure within 50 pounds per square inch above and 50 pounds per square inch below the critical pressure of the solvent.
6. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 30° F. below and 30° F. above the critical temperature of the solvent under a pressure within 50 pounds per square inch above and 50 pounds per square inch below the critical pressure of the solvent.
7. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 20° F. below and 20° F. above the critical temperature of the solvent under a pressure within 50 pounds per square inch above and 50 pounds per square inch below the critical pressure of the solvent.
8. Process according to claim 1 in which the oil solvent mixture is heated to a temperature within about 10° F. below and 10° F. above the critical temperature of the solvent under a pressure within 50 pounds per square inch above and 50 pounds per square inch below the critical pressure of the solvent.

9. In a process for treating heavy hydrocarbon oil with liquefied propane, the method of recovering the propane from the treated oil which comprises heating the propane-oil mixture to near the critical temperature of propane, whereby the oil and propane become substantially immiscible in one another, and separating the oil layer from the propane layer.

10. In a process for treating heavy hydrocarbon oil with a mixture of light hydrocarbons, the method of recovering the light hydrocarbons, from the treated oil which comprises heating the oil-light hydrocarbon mixture to a temperature between the critical temperatures of the several light hydrocarbons while maintaining a pressure sufficient to retain the light hydrocarbons in liquid phase, and separating the oil layer from the light hydrocarbon layer.

11. Process for treating heavy hydrocarbon oil which comprises diluting the oil with a light hydrocarbon whereby asphaltic bodies are caused to precipitate, removing the precipitated asphaltic bodies, chilling the remaining solution to a temperature at which wax is caused to separate, removing the wax, heating the wax-free solution to a temperature near the critical temperature of the light hydrocarbon, whereby the oil and light hydrocarbon become substantially immiscible in each other, and separating the oil layer from the light hydrocarbon layer.

12. Process according to claim 11 in which the light hydrocarbon comprises propane.

13. The method of separating a light hydrocarbon solvent from a mixture of the same with a heavy hydrocarbon oil, which comprises heating the oil-solvent mixture to a temperature at which the oil becomes substantially insoluble in the solvent and then separating the oil and solvent layers.

14. The method of separating liquefied propane from a mixture of the same with a heavy hydrocarbon oil, which comprises heating the propane-oil mixture to a temperature between the approximate limits of 180° F. and 204° F. to cause the mixture to separate into a propane layer and an oil layer and separating these two layers.

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