May 18, 1943 H. P. WICKHAM ET AL 2,319,381

CONTINUOUS DEWAXING

Filed June 15, 1940 2 Sheets-Sheet 1

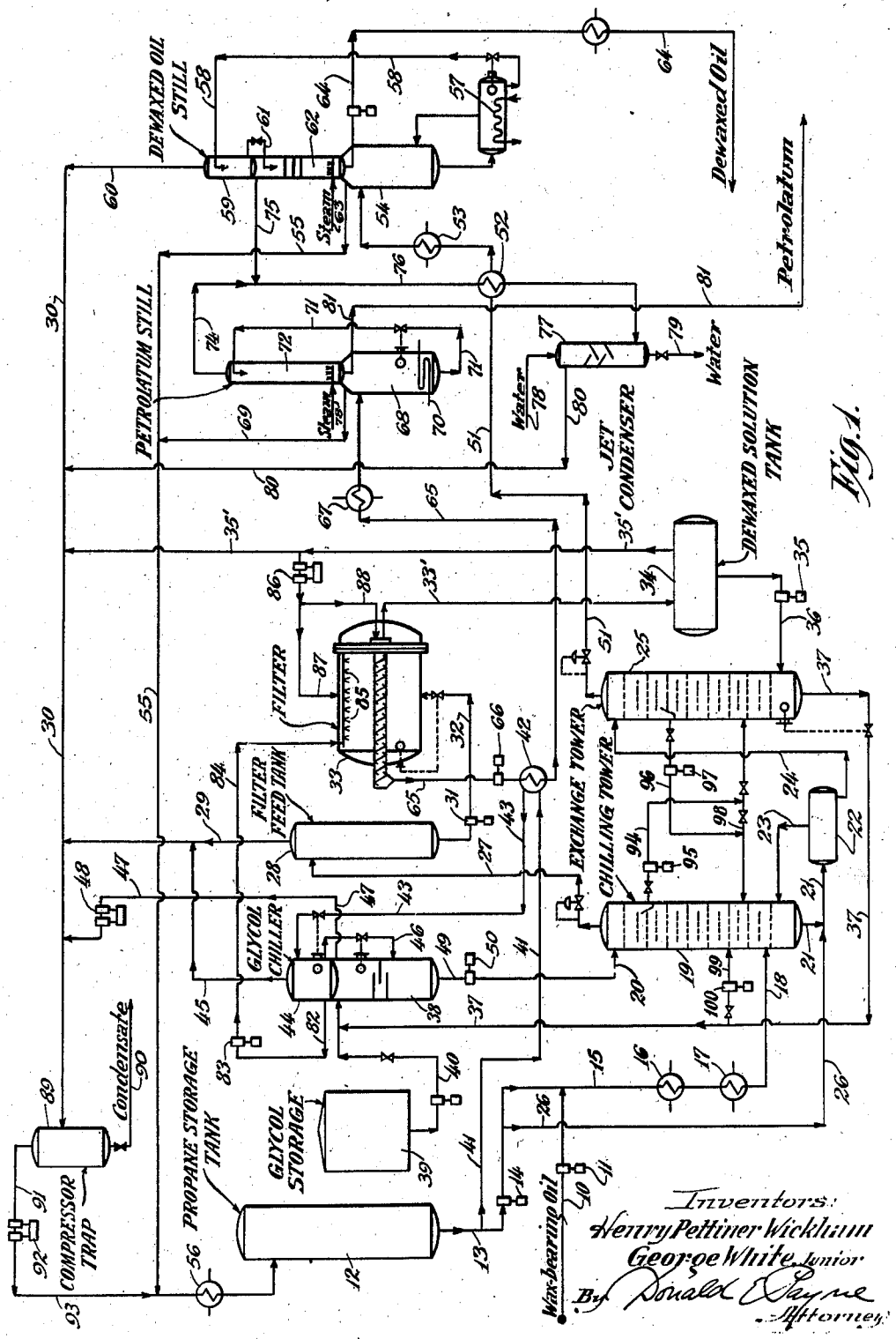


FIG. 1.

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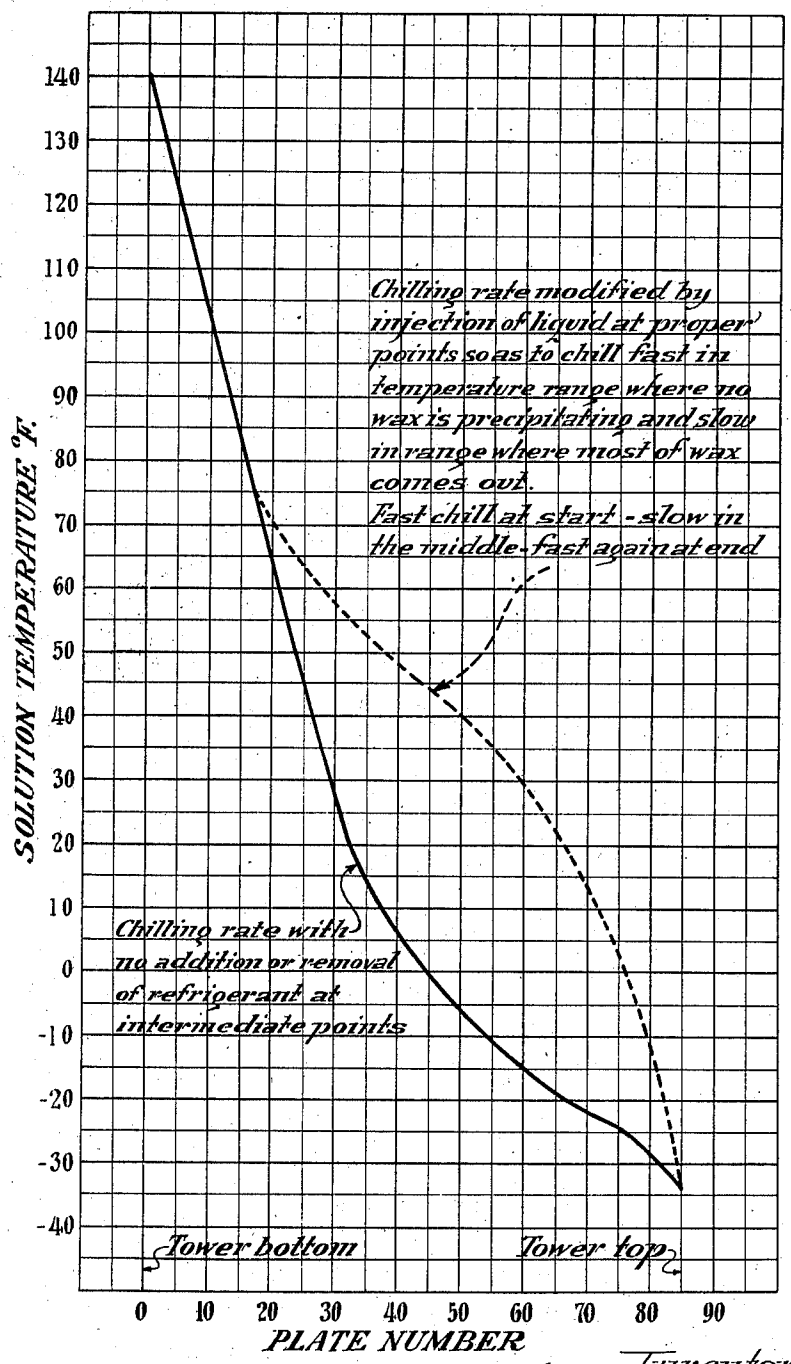


Fig. 2.

Inventors:
Henry Pettiner Wickham
George White, Junior
By Donald E. Payne
Attorney.

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CONTINUOUS DEWAXING

Henry Pettiner Wickham, New York, N. Y., and George White, Jr., Summit, N. J., assignors to Standard Oil Company, Chicago, Ill., a corporation of Indiana

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10 Claims. (Cl. 196—18)

This invention relates to the separation of wax from oils and it pertains more particularly to an improved system for utilizing either an immiscible or partially miscible refrigerant for effecting controlled continuous chilling prior to the wax separation step.

The U. S. patent to Brown 2,005,988 describes the use of a nonmiscible refrigerant in a propane dewaxing system. An object of our invention is to provide an economical commercial dewaxing process along a general line suggested by Mr. Brown. A further object is to solve the problem of emulsion difficulties which arises in any system using an intimate mixture of a lubricating oil with a nonmiscible or partially miscible refrigerant. A further object is to provide improved methods and means for controlling the crystallization of wax in the chilling step. A further object is to provide improved methods and means for chilling the refrigerant prior to admixing the chilled refrigerant with waxy oil solution.

A further object of the invention is to improve the quality of a lubricating oil while it is being continuously chilled for the wax separation step. A further object is to provide a continuous chilling system which may be employed for the dewaxing of oils in normally liquid diluents as well as in normally gaseous diluents.

An important object of the invention is to effect large savings in the investment and operating costs of a commercial dewaxing system and to provide a continuous chilling step which avoids the difficulties which have heretofore been encountered in the operation of scraped surface heat exchangers.

A further object is to provide a continuous chilling system employing normally gaseous diluents, which system will not be subject to the difficulties of flash chilling. Previous proposals for continuous chilling by direct vaporization of a normally gaseous diluent have offered practically insurmountable problems with regard to separating the diluent without undue agitation and with regard to actually controlling the crystallization of the wax in such form as to permit subsequent separation by filtration or otherwise. Other objects of the invention will be apparent from the following detailed description.

In practicing our invention relatively nonviscous waxy oils may be continuously chilled and dewaxed without the use of diluents but in most cases we prefer to employ a diluent for facilitating the crystallization of wax in separable form and for reducing the viscosity of the oil at wax separation temperatures. Our preferred diluent is propane or a mixture of propane with other normally gaseous hydrocarbons such as propylene and smaller amounts of butanes, butylenes, ethane and ethylene. Other normally gaseous diluents such as methyl ether, sulfur dioxide, etc., may be employed—in fact any diluent or mixture of diluents may be employed which exerts the desired effect on wax crystallization and low temperature oil viscosity and which is not chemically affected by or completely miscible with the refrigerant.

Instead of employing normally gaseous diluents we may employ normally liquid diluents such as gasoline, light naphthas, oleum spirits or other volatile hydrocarbons, ketones such as acetone and methyl ethyl ketone, alcohols such as propyl and butyl alcohols, ethylene dichloride and in fact any of the well-known antisolvents which have heretofore been employed in dewaxing processes. Where the antisolvent is not completely miscible with the oil at dewaxing temperatures we may employ miscibility agents such as benzol, toluol or the like.

As a refrigerant we may employ either a nonmiscible refrigerant or a partially miscible refrigerant and the selection of the refrigerant will, of course, depend to some extent on the nature of the diluent employed. When propane is the diluent we prefer to use an ethylene glycol-water solution as the refrigerant, other preferred examples being aqueous brines, diethylene glycol-water solutions, ethylene glycol monomethyl ether-water solutions, alcohols in their aqueous solutions, inorganic acids and bases, mercury, amino compounds, etc. Where a benzol ketone solvent is employed the refrigerant should, of course, be of such nature that it will not react or be miscible in any appreciable extent with either component of this solvent. Mercury can, of course, be employed with this or any other solvent.

Our invention makes possible an actual treating or refining of the oil during the chilling step. Thus if sulfur dioxide, sulfuric acid, furfural, dichlorethyl ether, or aqueous phenolic compounds (provided they have a low enough freezing point) are used as refrigerants they simultaneously effect the desired continuous cooling and effect a partial treating or refining of the oil which is being cooled. If such substances are employed as refrigerants, certain of them at least will be partially dissolved in the oil but it is a simple matter to recover such solvents from the dewaxed oil in the same way that solvents

are removed from oil in conventional solvent extraction processes.

Briefly, we first dilute the oil with the diluent or antisolvent and then countercurrently contact the oil in a chilling tower with the non-miscible or partially miscible refrigerant which has previously been chilled to a temperature of about —40° to —50° F. (when propane is employed as the diluent), the size of the tower, the rate of flow in the tower, etc., being so designed as to prevent emulsion difficulties and to provide the desired rate of cooling. The warm refrigerant leaving the base of the chilling tower is then countercurrently contacted with dewaxed oil solution in another countercurrent tower which we shall call the heat exchange tower, wherein the dewaxed oil solution is warmed to a temperature of about 130° F. and the refrigerant is cooled to a temperature of about —30° to —40° F. The refrigerant is then further cooled by exchange with flashed propane or by any other suitable refrigeration means to the original temperature which is preferably about —45° to —50° F. for reintroduction into the top of the chilling tower. A feature of our invention is the controlled chilling which we may effect in the chilling tower not only by carefully controlling the amounts and flow rates of oil and refrigerants in this tower but by introducing diluents or solvents or by introducing or withdrawing separate streams of refrigerants at one or more points along the tower. By this simple expedient we can carefully control the rate of cooling at any particular temperature and we can thus produce that type of wax crystallization which is so essential for the subsequent wax separation step. We prefer to effect the wax separation by filtration but it should be understood that other means such as settling, centrifuging and the like may be used.

The invention will be more clearly understood from the following detailed description and from the accompanying drawings which form a part of this specification and in which:

Figure 1 is a schematic flow diagram of a commercial 2000 barrel per day dewaxing plant embodying our invention, and

Figure 2 is a chart showing tower temperatures at various tower levels both with and without refrigerant injection and removal, thus illustrating modification of chilling rates.

The wax-bearing oil which may be either a distillate or residual stock is introduced through line 10 by pump 11 at the rate of about 2000 barrels per day. Propane from tank 12 (which may be at about 100° F. and about 200 pounds per square inch pressure) is withdrawn through line 13 by means of pump 14 at the rate of about 5000 barrels per day and mixed with the waxy oil charge in line 15. This mixture is heated in heat exchanger 16 to a temperature of about 150° F. in order to effect complete solution of both the wax and oil in the solvent—it is essential that this temperature be high enough to completely dissolve the wax in order that the previous wax crystallization tendencies will not carry over into the subsequent chilling step.

The mixture is then cooled with available condenser water in heat exchanger 17 after which it is introduced through line 18 at the base of chilling tower 19 which is maintained at a pressure of about 350 to 400 pounds per square inch. This tower is preferably about 9 feet in diameter and about 50 feet tall and is preferably provided with a large number of baffle plates in order to insure intimate contact between the counter flowing liquids. The propane-oil solution introduced near the base of the tower is preferably at a temperature of about 130° to 140° F.

The ethylene glycol-water solution is introduced through line 20 near the top of tower 19 at a temperature of about —48° F. and at the rate of about 3040 barrels per day. Either the oil solution or the refrigerant may constitute the continuous phase in tower 19 but since emulsions are formed during the direct contact of the two liquids and this emulsion tends to collect on the surface of the heavy refrigerant layer we prefer that the waxy oil solution be held in the continuous phase so that the inter-phase level will be maintained at the bottom or warm section of the tower where it will not interfere with the normal flow of the liquids. For this reason it is advantageous to hold the bottom of the chilling tower at a temperture of about 140° F., at which temperature the emulsion tends to break so that a clean stream of refrigerant may be removed from the tower. If there is insufficient breaking of the emulsion in the base of the tower we may withdraw the emulsion with the refrigerant through line 21 to settling tank 22 from which oil may be returned to the tower through line 23 and refrigerant passed by line 24 to the top of heat exchange tower 25. Additional diluent may be added to this withdrawn mixture through line 26 to facilitate separation and to thoroughly remove any dissolved or entrained wax. Such separation may also be facilitated by regulating the temperature of the liquids in settler drum 22.

In actual practice we have found that a large number of baffles effects great improvement in the contact and heat exchange obtainable in tower 19, about 50 to 100 baffles being preferred. Tower velocities must be kept low in order to prevent serious emulsion formation and in order to insure complete separation of the two phases. In practice we have used a superficial velocity of about 25 feet per hour for settling and about 125 feet per hour in the contact area when using the ethylene glycol solution as a refrigerant. The average chilling rate in the tower is preferably about 3° to 4° F. per minute but this chilling rate may be varied in different parts of the tower in order to effect the desired type of wax crystal formation, as will be hereinafter discussed.

The waxy oil solution leaves the top of chilling tower 19 at a temperature of about —43° to —45° F. and is withdrawn through line 27 to filter feed tank 28 which is vented through line 29 to low pressure propane line 30. Feed tank 28 is maintained at about atmospheric pressure and about —43° F. From this tank the slurry is forced by pump 31 thru line 32 to continuous filter 33 which may be of the type described in Keith et al. Patent 2,050,007. The filtrate is withdrawn from the filter through line 33' to dewaxed oil solution tank 34 which is likewise vented by line 35' to line 30.

The cold dewaxed solution from tank 34 is introduced by pump 35 in line 36 into the base of heat exchange tower 25. This heat exchange tower is about the same size but perhaps slightly smaller than tower 19 and should likewise be provided with baffles and carefully controlled as to flow rates. The dewaxed oil solution which contains about 5,440 barrels per day of propane and 1,740 barrels per day of dewaxed oil is introduced at the base of tower 25 at a temperature of about —43° F. and withdrawn from the top of the tower at a temperature of about 130° F., this

tower being maintained under a pressure of about 300 pounds per square inch. The refrigerant which is introduced at the top of the tower at 135° F. is withdrawn from the base of the tower at about —33° F. and passed by line 37 to glycol chilling tank 38 together with any makeup refrigerant that may be required from storage tank 39 and line 40.

The method of effecting this last increment of refrigerant chilling will vary with the use of different diluents but where propane is employed as the diluent we prefer to effect this chilling by merely cooling a portion of the propane and vaporizing this propane in contact with the glycol. About 2950 barrels per day of propane may be withdrawn from the storage tank through line 41, cooled in heat exchanger 42 to a temperature of about 65° F. and introduced by line 43 into evaporator 44 which is vented by line 45 to line 30. Vaporization of propane chills the remaining propane liquid to a temperature of —43° F. at which temperature a portion of it is introduced through line 46 into an intermediate or lower point of glycol chilling tank 38. This tank is maintained under partial vacuum so that the vaporization of propane therein results in a final refrigerant temperature of about —48° F., the propane vapors from this tank being withdrawn through line 47 to the suction side of booster compressor 48 which discharges into line 30. Refrigerant at —48° F. is pumped from chilling tank 38 to the top of chilling tower 19 through line 49 by pump 50.

The warm dewaxed oil solution leaving the top of tower 25 is passed through line 51, heat exchanger 52 and low pressure steam heater 53 into high pressure flash tower 54 from which the vaporized propane passes directly by line 55 to the condenser 56 and storage tank 12. The solution in the base of this high pressure flash tower is heated by high pressure steam coils 57 and the hot oil which is thus substantially depropanized is introduced by line 58 into atmospheric pressure flash drum 59 from which propane passes by line 60 to line 30. The oil from the atmospheric flash drum is then passed by line 61 into stripping column 62 wherein it is stripped by steam introduced through line 63. The final dewaxed oil is removed through line 64 to storage.

Wax from the filter is withdrawn through line 65 by means of pump 66 and passed through heat exchanger 42, low pressure steam heater 67 and high pressure flash drum 68 from which propane is passed directly through lines 69 and 55 to condenser 56 and storage tank 12. The base of tower 68 is heated by high pressure steam coils 70 and a substantially depropanized wax is introduced by line 71 into atmospheric flash and stripper tower 72, into which steam is introduced through line 73. Propane and steam from tower 72 are withdrawn through line 74 and admixed with the propane and steam withdrawn from stripper 62 through line 75, these combined streams being passed through line 76 and heat exchanger 52 to jet condenser 77 into which water is introduced through line 78 and withdrawn through line 79. Propane from the jet condenser is returned through line 80 to line 30. Petrolatum is removed from the base of tower 72 through line 81.

Cold propane for washing the filter cake is withdrawn from propane chilling tank 44 through line 82 by pump 83 and passed by line 84 to distributors 85 in the filter shell. The filter cake is dried by means of propane gases pumped into the filter shell from line 35' by means of compressor 86, the drying gases being introduced through line 87 and the blow-back gases for quick discharge being introduced through line 88.

Low pressure gas line 30 leads to compressor trap 89 from which water or condensed hydrocarbons may be withdrawn through line 90. Propane is taken from this trap through line 91 to the suction side of compressor 92 and the compressed propane is passed by line 93 to condenser 56 and storage tank 12.

A feature of the above-described system is the countercurrent contacting of cold refrigerant with warm oil solution for chilling that solution and warming the refrigerant and the subsequent countercurrent contacting of the warm refrigerant with cold dewaxed solution to warm the solution and chill the refrigerant. It should be particularly noted that in both of these steps sufficient pressure is maintained on the system to prevent vaporization of propane because such vaporization disturbs the equilibrium in the towers, causes agitation and disrupts the operation generally. Where a solvent such as naphtha or benzol ketone mixtures are employed it will, of course, not be necessary to operate these towers under pressure.

An important feature of our chilling system is the facility with which cooling rates may be regulated in various parts of the tower. By operating the system as hereinabove described the chilling curve in tower 19 is as shown in the solid line in Figure 2. Chilling is fairly rapid in the lower end of the tower but the chilling rate markedly falls off in the upper end of the tower due to the heat of fusion of the wax which is being solidified. It is desirable that the chilling rate be relatively slow in the temperature range wherein wax is being crystallized. Relatively rapid chilling rates are desirable from the inlet temperature of 140° F. to about 70° F. and from a temperature of about —10 or —20° F. to —45° F. In systems employing indirect heat exchangers this variation in chilling rates is difficult to accomplish and the slow chilling rate during wax crystallization necessitates the use of extremely large heat exchange areas due to the very small temperature differential which must be employed for obtaining the slow chilling. In accordance with our invention, however, the rate of chilling in different parts of the chilling tower may be easily regulated so that at the ends of the tower the chilling rate is very rapid while in the intermediate portion of the tower, preferably the major portion of the tower, the temperature differential between refrigerant and liquid may be very small and the chilling rate accordingly very slow.

Assuming that the temperature differential at the top of the chilling tower is 5° F., i. e., refrigerant inlet at —48° F. and oil outlet at —43° F., a rapid chilling rate may be obtained in the upper part of this tower, say to that point at which the temperature is —20° F. At that point in the tower a part of the cold glycol may be withdrawn through line 94 and passed by pump 95 to the lower part of tower 25. The removal of this cold refrigerant from the upper part of the tower is not essential but it may be desirable if slower chilling rates are desired in the intermediate tower section. Similarly, cold glycol at about 50° to 65° F. may be trapped out of tower 25, withdrawn through line 96 and introduced by pump 97 to that point in chilling tower 19 where the temperature is about 70° F. Thus in

the lower part of the tower where little or no wax is undergoing crystallization we will have a large temperature differential between refrigerant and waxy oil solution so that rapid chilling rates will be obtained. The temperature at which cold refrigerant is introduced into the chilling tower should not be low enough, of course, to produce harmful "shock chilling" at the point of introduction. Referring to Figure 2, this modified operation will throw us on the dotted line, i. e., will give us relatively low chilling rates in that range where wax is being crystallized and high chilling rates both above and below this range. In other words, the temperature differential of the refrigerant and waxy oil solution in the base of the tower may be about 5° to 20° F., in the top of the tower it may be about 3° to 10° F. while in the main portion of the tower it may only be about 1° to 3° F., preferably about 2° F. It should be understood that although we have disclosed one system for modifying chilling rates in tower 19 many other methods can be employed to accomplish this same purpose. Glycol from the upper part of the tower may be introduced into the lower part of the tower through line 98. Glycol from line 37 may be introduced into the lower part of the tower through line 99 by means of pump 100. Glycol from various points of the exchanger tower may be introduced at various points in the chiller tower in order to provide conditions in the chilling tower to give any desired chilling rate in any particular temperature range. This simple but very effective method of controlling chilling rates in a continuous chilling system is of great practical importance.

Another method of controlling the rate of chilling in the tower is to add diluent, for instance at that point in the tower where the solution has been chilled to about 70° F. This addition of diluent changes the ratio of wax-bearing oil to diluent which, under many circumstances results in more desirable forms of wax crystallization. The wax solution step, for instance, may be effected with a 1:1 diluent ratio and one or more volumes of diluent may be added to the chilling tower after the initial solution has been rapidly chilled to that temperature at which wax begins to separate out.

Where the refrigerant or diluent is of such nature that it effects a partial extraction or treating of the oil the apparatus will, of course, have to be slightly modified. For example, if sulfuric acid is employed as a refrigerant the apparatus will have to be of acid resistant material and provision will have to be made for removing spent acid sludge and for neutralizing the oils and gases which may contain acidic materials. The acid from the chilling tower may, for instance, be introduced into a settling tower and washed with propane or other diluent for the removal of oil and sludge therefrom prior to its reintroduction at the top of the heat exchange tower. The oil and sludge which is removed from the acid together with a portion of the acid may then be mixed with the incoming oil charge and introduced into a settling tank from which spent acid sludge is withdrawn at the base and partially treated oil charge, together with recovered oil, is taken from the top for reintroduction at the base of the chilling tower.

Should partially miscible refrigerants such as conventional selective solvents be employed, the recovery systems will, of course, be slightly modified to provide for the extract material, the diluent from the extract, raffinate and wax being returned to tank 12 and the solvent or refrigerant being returned from these recovery systems to tank 39. When treating agents or selective solvents are employed, either all or a part of the warm solution from line 24 may be charged to a still for removing extract material from the selective solvent before the solvent is condensed and introduced into the top of exchanger tower 25, as has been hereinabove described.

While we have described in detail a preferred embodiment of our invention it should be understood that the invention is not limited to this particular embodiment nor to the temperatures, pressures and other operating conditions recited in connection with that embodiment. The application of our invention to the use of other solvents and to other miscible and partially miscible refrigerants will be apparent from the above description of our preferred embodiment. Instead of employing a propane flash chilling system for obtaining the last increment of refrigeration, the dewaxed oil solution may be further chilled to low temperatures and introduced into the base of exchanger tower 25 at such a low temperature that the refrigerant leaving the base of tower 25 may be introduced directly into the top of chilling tower 19. Other modifications and alternatives will be apparent to those skilled in the art from the above disclosure.

We claim:

1. The method of controlling the chilling rate in a countercurrent system wherein a warm waxy oil solution is countercurrently and directly contacted with a cold normally liquid refrigerant which comprises withdrawing refrigerant from the system near the low temperature end thereof, and adding refrigerant to said system adjacent the high temperature end thereof, the temperature of the added refrigerant being lower than the temperature of the refrigerant in the countercurrent system at the point of addition.

2. The method of operating a countercurrent chilling tower for continuously cooling a waxy oil solution in order to solidify the wax in separable form, which method comprises introducing cold normally liquid refrigerant at the top of the tower, introducing a warm waxy oil solution at the base of the tower, countercurrently and directly contacting said refrigerant with said oil solution in said tower and maintaining a higher temperature differential between the refrigerant and the oil solution in the lower part of the tower than is maintained in the intermediate part of the tower by introducing additional cold refrigerant liquid into the countercurrent stream in the lower part of the tower.

3. The method of claim 2 which includes the further step of maintaining a higher temperature differential between the refrigerant and the oil solution in the upper end of the tower than is maintained in the intermediate section of the tower by removing refrigerant liquid from the countercurrent stream in the upper part of the tower.

4. The method of dewaxing oil which comprises heating about 2 volumes of waxy oil with about 5 volumes of propane to a sufficiently high temperature to effect complete solution of both wax and oil in the propane, bringing said solution to a temperature of about 140° F., countercurrently and directly contacting the cooling solution in a

countercurrent tower with a normally liquid refrigerant which is not completely miscible with said solution, maintaining sufficient pressure in said tower to prevent vaporization of propane, introducing refrigerant at the top of the tower at a temperature of about —40° to —50° F. mechanically separating wax from the waxy oil mixture leaving the top of the tower, countercurrently contacting the dewaxed oil solution with refrigerant leaving the bottom of the tower whereby said refrigerant is partially cooled, maintaining a sufficient pressure in this last-named countercurrent contacting step to prevent vaporization of propane, further chilling the refrigerant to a temperature of about —40° to —50° F., by the addition of cold propane thereto and the vaporization of propane therefrom and returning the cold refrigerant to the top of said tower.

5. The method of dewaxing oil which comprises diluting a waxy oil with propane, countercurrently and directly contacting said diluted oil with a cold normally liquid refrigerant which is not completely miscible therewith whereby the refrigerant is heated and the waxy oil solution is cooled to effect solidification of wax, removing the cooled wax-diluted oil mixture from the refrigerant and mechanically separating the wax from said mixture to obtain a dewaxed diluted oil, countercurrently and directly contacting the dewaxed diluted oil with warm refrigerant from said first named countercurrent contacting step whereby the refrigerant is partially cooled, finally cooling the partially cooled refrigerant to a temperature lower than the dewaxing temperature, returning the finally cooled refrigerant to said first named countercurrent contacting step, and maintaining a pressure in both countercurrent contacting steps which is sufficiently high to prevent substantial vaporization of propane therein.

6. The method of dewaxing oil which comprises directly and countercurrently contacting said oil with a cold normally liquid refrigerant which comprises sulfuric acid and which is not completely miscible with said oil whereby the refrigerant is heated and the oil is cooled to effect solidification of wax, removing the cooled wax-oil mixture from the acid refrigerant and mechanically separating the wax from the removed mixture to obtain a dewaxed oil, countercurrently and directly contacting the dewaxed oil with warm acid refrigerant from said first named countercurrent contacting step whereby the acid refrigerant is partially cooled, finally cooling the partially cooled acid refrigerant to a temperature lower than the dewaxing temperature and returning the finally chilled acid refrigerant to said first named countercurrent contacting step.

7. The method of dewaxing oil which comprises countercurrently and directly contacting said oil with a cold normally liquid refrigerant which is not completely miscible therewith whereby the refrigerant is heated and the oil is cooled to effect solidification of wax, introducing additional refrigerant at an intermediate point in said contacting step, removing the cooled wax-oil mixture from refrigerant with which it has been contacted and mechanically separating the wax from said mixture to obtain a dewaxed oil, countercurrently and directly contacting the dewaxed oil with warm refrigerant from said first named countercurrent contacting step whereby the refrigerant is partially cooled, finally cooling the partially cooled refrigerant to a temperature lower than the dewaxing temperature and returning the finally cooled refrigerant to said first named countercurrent contacting step.

8. The method of operating a continuous chilling system for the dewaxing of oil which comprises countercurrently and directly contacting a waxy-oil solution in a contacting zone with a normally liquid refrigerant which is not completely miscible therewith, introducing the waxy-oil solution at one end of said zone at a temperature of about 140° F., introducing the refrigerant at the other end of the contacting zone at a temperature of about —50° F., maintaining the waxy-oil solution in the continuous phase throughout the major part of said zone, separating waxy-oil from refrigerant at one end of said zone and separating refrigerant from waxy-oil at the other end of said zone, and maintaining a superficial velocity of the waxy-oil solution in said zone within the approximate range of 25 to 125 feet per hour.

9. The method of claim 8 which includes the further step of adding additional refrigerant at an intermediate point of said zone, the temperature of the added refrigerant being lower than the temperature in the zone at the point of additional refrigerant introduction.

10. The method of dewaxing oil which comprises countercurrently and directly contacting said oil with a cold normally liquid refrigerant which is not completely miscible therewith in a countercurrent contacting zone whereby the refrigerant is heated and the oil is cooled to effect solidification of wax, maintaining one end of said zone at a temperature of at least about 100° F. and the other end of said zone at a temperature not substantially higher than about —10° F., introducing additional cold refrigerant into said zone at a point at which the temperature of the oil is within the general vicinity of 70° F., removing cooled oil-wax mixture from the low temperature end of said zone, mechanically separating the wax from said mixture to obtain a dewaxed oil, countercurrently and directly contacting the dewaxed oil with warm refrigerant from the first named countercurrent contacting step whereby the refrigerant is partially cooled, finally cooling the partially cooled refrigerant to a temperature lower than the dewaxing temperature, and returning the finally cooled refrigerant to said first-named countercurrent contacting step.

HENRY PETTINER WICKHAM.
GEORGE WHITE, JR.