Oct. 14, 1952 K. H. WANDERER ET AL 2,614,065

PROPANE DEWAXING CHILLING PROCEDURE

Filed March 30, 1950

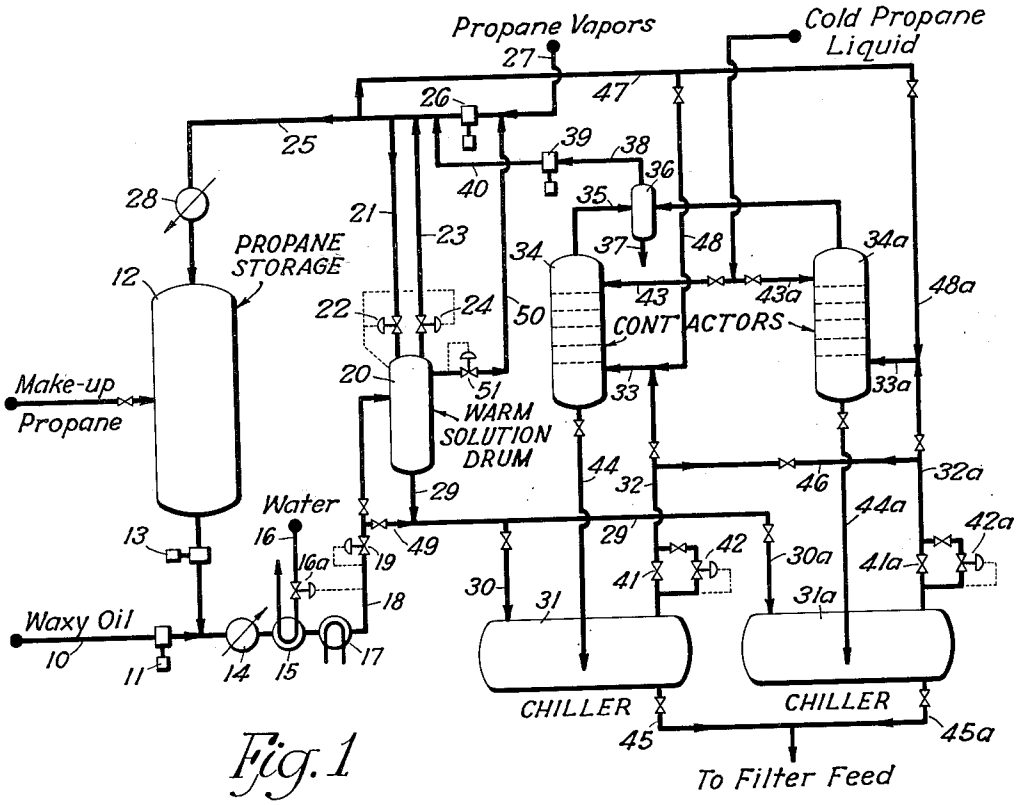


Fig. 1

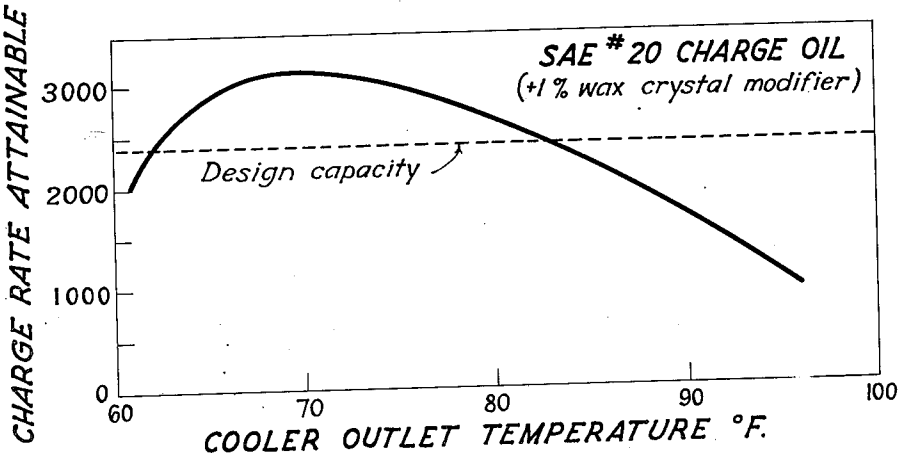


Fig. 2

INVENTORS:
Kenneth H. Wanderer
Leonard A. Hays, Jr.
Robert B. Selund
BY Donald E. Payne
ATTORNEY

Patented Oct. 14, 1952 2,614,065

UNITED STATES PATENT OFFICE

2,614,065

PROPANE DEWAXING CHILLING PROCEDURE

Kenneth H. Wanderer, Chicago, Ill., and Leonard A. Hays, Jr., Hammond, and Robert B. Selund, Highland, Ind., assignors to Standard Oil Company, Chicago, Ill., a corporation of Indiana

Application March 30, 1950, Serial No. 152,824

17 Claims. (Cl. 196—18)

1

This invention relates to an improved chilling procedure for propane dewaxing and it pertains more particularly to an improved method and means for improving the filterability of chilled propane-oil-wax slurries.

Propane dewaxing is a known operation in extensive commercial use for separating wax from lubricating oil stocks, a commercial plant for such operation being generally described for example in U. S. 2,143,872. An object of this invention is to provide an improved method of chilling a propane-waxy-oil solution so that the final resulting propane-oil-wax slurry will be more readily filterable; by increasing the filterability of such slurry, the throughputs in a given commercial installation of limited filter capacity may be greatly increased. A further object is to simplify and expedite the chilling procedure. A further object is to provide a method of cooling which will eliminate the necessity of employing a warm solution tank for charging chillers. Other objects will become apparent as the detailed description of the invention proceeds.

Heretofore it has usually been considered essential in commercial propane dewaxing systems to avoid shock chilling. Commercial units are provided with a warm solution tank large enough to hold a chiller charge. The warm solution tank is maintained at a temperature above the cloud point of the propane-oil-wax solution. Usually each chiller is warmed up to a temperature above wax precipitating temperatures before the charge is introduced thereto and even in those exceptional cases where the chiller is initially not warmed up to so high a temperature, the temperature of the chiller immediately after the filling step and before the initiation of evaporated cooling approximates very closely the temperature which is maintained in the warm solution tank. We have found that a remarkable improvement in propane-oil-wax slurries can be obtained by purposely flash chilling, i. e. shock chilling, the propane-waxy-oil solution (preferably with added pour point depressor) from a temperature approximately 10° F. above its visual cloud point, through a temperature range of at least 5° F. but not more than 25° F. and avoiding re-solution of minute crystals which are thus produced, conventional cooling methods being applicable both before or after this flash chilling or shock chilling operation. Prior to the flash chilling step, the original hot waxy-oil solution should be cooled to a temperature which is at least about 2° F. and preferably about 10° F. above its visual cloud point; the method of cooling employed from the initial high solution temperature down to this temperature just above the cloud point is not critical and any known chilling methods may be employed.

2

When the propane-waxy-oil solution has reached a temperature about 10° F., or within the range of about 3° F. to 20° F., above its visual cloud point, it is shock chilled by being passed through a controlled pressure reducing valve into a zone of lower pressure which varies somewhat with particular charging stocks. For Mid-Continent SAE 10 stocks (visual cloud point 48° F.), the down-stream pressure should be about 85 p. s. i. g., for SAE 20 stocks (visual cloud point 60° F.) about 90 p. s. i. g., and for SAE 40 and heavier stocks (visual cloud point 70° F.) about 120—125 p. s. i. g. The flashing of propane which takes place immediately beyond the pressure reducing valve shock chills the propane-waxy-oil solution to a temperature which is sufficient to initiate wax crystallization, the extent of the temperature drop being in the range of about 5° to 25° F. and usually being sufficient to chill the solution to approximately, or slightly below, the visual cloud point. The visual cloud point may be determined by placing an aliquot portion of the solution in a glass capillary tube, solidifying the solution by chilling, sealing the tube containing solidified sample, then heating the sample to 150° F. to destroy all previous crystal formation and cooling the sample at approximately 5° F. per minute, noting the temperature at which the first visible wax crystal formation can be seen. The presence of pour test depressors has practically no effect on this visual cloud point but the visual cloud point may be about 5° to 10° F. lower than the temperature at which wax nuclei actually begin to form as indicated by known tests other than visual observation.

The shock chilled oil is preferably maintained at a temperature which is just below its actual cloud point and approximately, or slightly below, its visible cloud point until the chiller is filled; the temperature may vary a few degrees during the filling step but at no time should it be more than 15° F. below the visual cloud point.

The flash chilling or shock chilling is effected by a sharp pressure drop which is usually in the range of about 20 to 50 p. s. i. g. Thus, the initial cooling to a temperature approximately 10° above the visual cloud point may be under a pressure of approximately 500 p. s. i. g. in order to insure liquid phase conditions in a heating

step which precedes initial cooling. After cooling to about 10° F. above the visual cloud point, the pressure may be reduced to approximately 150 p. s. i. or lower without any flashing of propane but the last increment of pressure drop (down to about 120 p. s. i. g. for SAE 40 stocks or about 85 p. s. i. g. for SAE 10 stocks) which is usually in the range of 20 to 50 p. s. i. g. effects the shock chilling which apparently causes the formation of wax nuclei which have no tendency to settle out of the solution.

When the chiller charge is all introduced, such charge is then cooled by controlled evaporation of propane in the usual manner; for example, it may be cooled from about 50° F. to −45° F. in approximately 15 to 20 minutes. The chilled slurry is then ready for filtration although it may be first introduced into a filter feed tank and the last portion of the cooling may be effected in the filter feed tank.

When employed in an existing propane dewaxing plant equipped with a warm solution tank, we prefer to operate with substantially no liquid level in the warm solution tank but to merely use it as an enlarged portion of the transfer line to take care of any surge when valves are shifted. A chiller, which has just been emptied and which is about −45° F., is first warmed up by condensation of propane vapors introduced from the other chiller and/or from the compressor discharge. This warm-up operation only requires about one-half to one minute and it is terminated when the pressure in the chiller reaches the desired down-stream pressure which, as above stated, is in the range of about 80 to 125 p. s. i. g., and is about 90 p. s. i. g. for SAE 20 stocks. The propane charge stream leaving the pressure reducing valve is then switched from the chiller just filled to the chiller which has been warmed up to a temperature just below the cloud point of the entering stream and the stream flows directly into the chiller at a substantially constant temperature, the propane vaporized in the flash chilling step being condensed in or vented from the chiller which is continuing to warm up so that at the end of the filling operation, the chiller vessel is at substantially the same temperature as the solution contained therein which in turn is at a temperature just below its actual cloud point. The warm-up, filling and emptying of one chiller thus requires the same time as the evaporating cooling in the other chiller.

Modifications in the existing plants are desirable in order to obtain best results in the practice of our invention. If it is desired to employ warm solution tanks so that each chiller may be filled almost instantaneously, said tank serves the additional function of serving as a shock chilling tank by providing a line from the upper part of this tank to the holding compressor inlet and providing said line with a valve for automatically holding the desired pressure (90 p. s. i. g. in the case of SAE 20 stock) in the warm solution tank. However, the warm solution tank may be entirely dispensed with if the feed lines to the chiller are sufficiently large to accommodate any surge during shifting of valves. When the warm solution tank is entirely dispensed with, the desired down stream pressure is either manually or automatically controlled in the vapor line leading from the chiller itself so that any flashed propane which is not condensed to effect further warming up the metal chiller vessel is withdrawn from the chiller.

Usually a wax crystal modifier of the pour point depressor type is added to the waxy-oil solution prior to chilling. When the solution is shock chilled from a temperature just above its visual cloud point through a range of at least 5°, but not more than 25° F., to a temperature just below its actual cloud point, it appears that wax nuclei are formed which have no tendency to settle out in the chiller but which on the other hand remain suspended throughout the charging solution in the chiller so that in the subsequent evaporative cooling step wax crystals are formed which are of greater regularity and which can be filtered much faster, not only giving much more rapid filter rates, but providing better filter cakes from the standpoint of cake washing and handling and at the same time giving a dewaxed oil solution which, on removal of propane, will have the desired low cold test. In the absence of our shock chilling step in the specified range, the wax crystals tend to be more flat and platelike so that they pack on the filter; when our improved technique is employed, the wax crystals are not only of more uniform size, but they have the appearance of rough irregularly shaped crystals tending to be needles radiating from central nuclei. By our method of shock chilling from just above to just below the cloud point, we have increased the capacity of a commercial propane dewaxing plant from its design capacity of about 2400 barrels per stream day to over 3000 barrels per stream day, increasing the filter rate from about 3.7 to over 4.6 gallons of charge per hour (based on incoming charge) per square foot of filter surface.

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

Figure 1 is a schematic flow diagram of that portion of the propane dewaxing system to which our invention relates, and

Figure 2 is a chart illustrating the increase in charge rate capacity for a commercial plant which has been accomplished by the practice of our invention.

Our invention is applicable to waxy-oil stocks generally, but is particularly advantageous for the dewaxing of lubricating oil stocks of the SAE 10 to SAE 50 range, as well as bright stocks. These lubricating oil stocks may be untreated distillates from high quality crudes or may be distillates which have been subjected to acid treating, solvent extraction, or other known refinery processes. The invention also may be applicable to the destearinizing of vegetable and animal oils, fats, fatty acids, etc., and in fact to the separation of any crystallizable solids from oleaginous materials. In the following example, we will describe the dewaxing of an SAE 20 lubricating oil stock produced as a phenol extraction raffinate from a fraction of Mid-Continent crude. While describing the operating procedure, we shall give a general description of that portion of the propane dewaxing plant to which this invention relates.

Referring to Figure 1, the waxy-oil charge is introduced from source 10 by pump 11 and if it is not already hot, it is preheated to a temperature of the order of 150 to 220° F., e. g. about 200° F. Propane from storage tank 12, which is operated under sufficient pressure so that it is approximately 100° F. is withdrawn by pump 13 and admixed with the heated waxy-oil to give a propane-to-oil ratio which is usually in the

range of 1.5 to 2.5, e. g. about 1.8. In order to insure complete solution, the mixture is heated in steam exchanger 14 to a temperature in the range of about 140 to 200° F., e. g. about 180° F., after which it is passed through water cooler 15 through which cooling water is circulated through pipe 16 in amounts controlled by valve 16a. In warm weather the water-cooled solution may be passed through heat exchanger 17 and further cooled with cold filtrate, although exchanger 17 may be by-passed in cold weather. The extent of cooling is controlled by valve 16a in accordance with the temperature of the cooled solution in line 18 and since the visual cloud point of this particular solution is 60° F., the temperature in line 18 is held at 70° F. by temperature control valve 16a. Up to this point, the solution is under a relatively high pressure of about 500 p. s. i. g., which is controlled by valve 19.

In previous operations there was no flashing of propane as a solution passed through valve 19 to warm solution drum or tank 20 because this drum was maintained under a relatively high pressure by introducing propane vapors through line 21 and pressure control valve 22, any excessive pressure being avoided by discharging gases through line 23 in amounts controlled by pressure control valve 24, lines 21 and 23 being connected to the discharge line 25 of holding compressor 26, the inlet 27 of which returns propane from various parts of the dewaxing system for compression and condensation in condenser 28. With this existing system, valves 22 and 24 remain closed in our operation and no liquid level is carried in the warm solution drum. On the contrary, drum 20 is operated at a low pressure of about 90 p. s. i. g. by virtue of the fact that it is connected through lines 29 and 30 to chiller 31 which at this point is held at said pressure of 90 p. s. i. g. The solution from line 18, which is at about 70° F., is flash chilled as it leaves valve 19 to this region of lower pressure, thus effecting a shock chilling through a range of at least about 5° but not more than 25° F., e. g. to a temperature as much as 10° below the visual cloud point of the solution. The flash chilled solution together with the vapors resulting from the chilling flow together through substantially emptied drum 20 and lines 29 and 30 to chiller 31 so that chiller 31 accumulates a chilling charge in approximately 15 to 17 minutes. Some of the flashed propane condenses in chiller 31 and thus serves to bring the metal walls of the chiller to the same temperature as the waxy solution in the chiller; any additional propane vapors are discharged through lines 32 and 33 to propane contactor 34, the vapors from this contactor being withdrawn by line 35 to trap 36 for eliminating any condensate 37 and then passed by line 38 to chilling compressor 39 which discharges through line 40 to holding compressor discharge line 25. During the accumulation of charge in chiller 31, the pressure is maintained at about 90 p. s. i. g. by manually controlled valve 41, or preferably by completely closing valve 41 and employing a pressure control valve 42 for automatically holding the stated chiller pressure.

When the chiller has been charged to the desired extent, the alternate chiller 31a will have been chilled, emptied and reheated to about 50° F. so that the incoming charge can immediately be transferred from line 30 to line 30a. The cooling of the chiller charge from about 50° to —45° F. is effected by opening valve 41 and withdrawing propane vapors through lines 32 and 33, contactor 35, trap 36 and line 38 to compressor 39. During the chilling, cold propane is added to contactor 34 through line 43 and this cold propane is added from the base of the contactor through line 44 to a low point in the chiller in amounts not only to compensate for propane lost by vaporization, but to increase the propane to oil ratio by about .5 so that the final chilled slurry will have a propane to oil ratio of the order of about 2.3. This chilled mixture which may be at about —30 to —45° F. is withdrawn through line 45 either to a filter or to a filter feed tank; when a filter feed tank is employed a portion of the cooling may be effected by vaporization of propane in the feed tank.

The filtration and propane recovery steps are substantially the same as those heretofore known to the art and, therefore, require no further description. As soon as chiller 31 is emptied, it is warmed up by condensing propane vapors which are being withdrawn from chiller 31a through line 41a by opening cross-over line 46 and allowing propane to pass through lines 32 and 33 to contactor 34 and thence through line 44 to chiller 31 (the valve in cold propane line 43 being closed). Alternatively or in addition, propane vapors from compressor discharge line 25 are introduced by lines 47, 48 and 33 to contactor 34 and thence introduced by line 44 to chiller 31. This warm-up only requires about one-half minute to one minute and the extent of warm-up is controlled by regulating valve 41 or setting valve 42 so that the pressure in the chiller cannot exceed 90 p. s. i. g. At this point, the condensed propane in chiller 31 will be at about 50° F., at which time it is ready to receive additional charge. The contactor and connecting lines for chiller 31a are similar to those hereinabove described in connection with chiller 31 and are hence shown by similar reference characters supplemented by the letter "a." Chilling in this example requires about 20 minutes and is balanced by about 2 minutes for emptying the other chiller, one-half to one minute for warming it up, and about 17 to 17½ minutes for introducing the next charge.

In the operation hereinabove described, it will be noted that warm solution tank 20 does not function as a warm solution tank, but merely as a portion of line 29. The system may thus be greatly simplified by eliminating the warm solution tank drum 20 altogether and simply passing the flash solution directly from valve 19 through line 49 to line 29, these lines being of sufficient size to take up any surge or accumulation of charge due to time required for valve shifting. On the other hand, if it is desired to accumulate chiller charges and to supply each chiller charge more rapidly, a connecting line 50 should be connected preferably to the holding compressor inlet line 27 and a pressure control valve 51 should be provided in line 50 so that the pressure in drum 20 is in this case maintained at about 90 p. s. i. g. The shock chilled charge may thus accumulate in the warm solution tank but the accumulator charge is not in this case a true solution but is a solution which contains dispersed wax nuclei formed in the flash chilling or shock chilling of the solution as it leaves valve 19.

In Figure 2 we have shown the remarkable effectiveness of our cooling procedure in increasing the charge rate to a commercial propane dewaxing plant. Without our feature of shock chilling from just above to just below the cloud point, this

plant could not by conventional chilling methods produce wax slurries which were sufficiently filterable to meet its design capacity of 2400 barrels per day. By cooling to 70° F., and then continuously flash chilling to about 50° F. (i. e. to a zone held at about 90 p. s. i. g.), the filterability of the propane-wax-oil slurry was increased so markedly that the charge rate could be increased to over 3000 barrels per stream day. From the chart, which is based on SAE 20 charge oil containing 1% of pour point depressor (Pourex), it will be seen that the temperature immediately prior to flash chilling must be at least 62, but not more than about 83° F. in order to even meet design capacity. When the temperature immediately prior to flash chilling was in the range of 3° to 20° F. above the visual cloud point temperature (60°), the plant capacity was increased by 200 barrels per day. When the solution temperature immediately prior to flash chilling was approximately 10° higher than the visual cloud point, maximum plant capacity was achieved which was more than 25% higher than design capacity. This chart graphically illustrates the remarkable increase in wax filterability which may be effected by the practice of our invention.

As heretofore stated, it is conventional practice to employ a pour point depressor with incoming charging stock to serve as a wax crystallization modifier. These pour point depressors may be petrolatum, crude residuum or fraction thereof, but this pour test depressor is preferably a chlorinated wax-naphthalene condensation product of the type generally known as Pourex and Paraflow used in amounts of the order of .01 to 2%, preferably about .1 to 1%. Polymerized methacrylate, marketed under the trade name Acryloid 150, has been found to be effective in somewhat lower amounts. No invention is claimed per se in the use of such known pour test depressors as wax crystallization modifiers since both the pour test depressors themselves and their effectiveness as wax crystallization modifiers in various concentrations are well known to the art and require no further description.

While the above example is based on the use of SAE 20 lubricating oil charging stock, it should be understood that the invention is likewise applicable to other lubricating oil stocks and to stocks of other viscosities. Thus, with an SAE 10 stock with a visible cloud point of about 48° F., the solution should be cooled immediately before the flashing step to a temperature in the range of about 50 to 65° F., preferably 55 to 60° F., and the zone down-stream of the flash chilling should be maintained at a pressure of about 85 p. s. i. g., thus giving a warm-up temperature of the order of 40 to 45° F. On the other hand, with SAE 40 stock having a visual cloud point of 70° F., the temperature immediately prior to flash chilling should be of the order of about 75 to 90° F. and the zone down-stream of the flash chilling step should be at a pressure of about 120 to 125 p. s. i., thus giving a warm-up temperature of about 50 to 60° F. It is not essential in all cases that the temperature down-stream of the flash chilling be maintained at all times below the visual cloud point since the latter portion of the charge may be introduced with little if any flash chilling and the final chiller charge may be slightly above the visual cloud point. This is particularly true when the flash-chilled solution is introduced directly into the chiller because any wax nuclei formed will not be redissolved in the short time between the completion of the charge and the initiation of evaporative cooling. For best results, the wax crystal nuclei formed by the flash chilling step should not be redissolved and, therefore, the chiller after charging should be at a temperature which does not substantially exceed, and which is preferably slightly below, the visual cloud point of the charging stock solution.

From the foregoing description, it will be apparent that we have accomplished the objects of our invention, and while we have described specific apparatus and operating conditions, it should be understood that many alternatives for same will be apparent from the above description to those skilled in the art. The term "propane" should be given the interpretation which is commonly afforded to this term in connection with dewaxing.

We claim:

1. The method of chilling a propane-oil-wax solution to obtain a wax slurry of improved filterability, which method comprises reducing the temperature of said solution to a point which is slightly above the visual cloud point of the solution, then flash chilling said solution through a temperature range at least about 5° F. but not more than about 25° F. by passing it instantaneously to a zone of lower pressure which pressure is sufficient to maintain the solution at a temperature slightly lower than its actual cloud point whereby the solution is shock chilled through the defined temperature range, and further chilling said solution by evaporative cooling to a wax filtration temperature.

2. The method of claim 1 which includes the further step of adding cold propane to the solution during the evaporative cooling in amounts sufficient to increase the propane-to-oil ratio of said solution.

3. The method of claim 1 wherein the flash chilling of the solution is effected when the temperature of the solution is reduced to a temperature in the range of about 3° to 20° F. above the visual cloud point of the solution.

4. The method of claim 1 wherein the flash chilling of the solution is from a temperature in the range of about 3° to 20° F. above the visual cloud point of the solution to a temperature below the visual cloud point of the solution.

5. The method of chilling a propane solution of a lubricating oil charging stock of about SAE 20 grade with a visual cloud point of about 60° F., which method comprises admixing said charging stock with propane to give a propane-to-oil ratio in the range of about 1.5:1 to 2.5:1, heating the mixture to obtain complete solution of the wax in the propane-to-oil solution, cooling the heated solution to a temperature in the range of about 62° F. to 83° F. while retaining it under the pressure required for maintaining propane in liquid phase in the heating step, then flash chilling the solution by passing it quickly to a low pressure zone which is maintained at approximately 90 pounds per square inch gauge and subsequently cooling said flash chilled solution by evaporative cooling to a wax filtration temperature.

6. The method of chilling a propane solution of a lubricating oil charging stock of about SAE 10 grade with a visual cloud point of about 48° F., which method comprises admixing said charging stock with propane to give a propane-to-oil ratio in the range of about 1.5:1 to 2.5:1, heating the mixture to obtain complete solution of the wax in the propane-oil solution, cooling the heated solution to a temperature which is in the range of about 50° to 70° F. while retaining it under the pressure required for maintaining propane in

liquid phase in the heating step, then flash chilling the solution by passing it quickly to a low pressure zone which is maintained at approximately 80 to 85 pounds per square inch gauge and subsequently cooling said flash chilled solution by evaporative cooling to a wax filtration temperature.

7. The method of chilling a propane solution of a lubricating oil charging stock at least as heavy as about SAE 40 grade with a visual cloud point of about 70° F., which method comprises admixing said charging stock with propane to give a propane-to-oil ratio in the range of about 1.5:1 to 2.5:1, heating the mixture to obtain complete solution of the wax in the propane-oil solution, cooling the heated solution to a temperature in the range of about 72° F. to 93° F. while retaining it under the pressure required for maintaining liquid phase in the heating step, then flash chilling the solution by passing it quickly to a low pressure zone which is maintained at approximately 120 to 125 pounds per square inch gauge and subsequently cooling said flash chilled solution by evaporative cooling to a wax filtration temperature.

8. In a process for dewaxing a lubricating oil stock wherein the said stock is admixed with propane, heated to obtain complete solution, cooled to low temperature for obtaining a slurry and filtered to remove wax from said slurry, the improvement which comprises effecting the chilling to a temperature in the range of about 2° to 23° F. above the visual cloud point of the solution while retaining the solution under a pressure which is greater than the vapor pressure of the propane at said temperature, then flash chilling said solution by passing it quickly to a lower pressure zone at which the pressure is in the range of about 80 to 125 pounds per square inch gauge and is sufficiently low to effect a shock chilling of said solution through a temperature range of at least about 5° F. but not more than about 25° F., and effecting the remainder of the cooling by vaporizing propane from the solution while adding cold propane to the solution to at least compensate for propane removed therefrom by evaporation.

9. The method of claim 8 which includes the additional step of effecting said chilling in the presence of an effective amount of a pour point depressor for modifying wax crystal structure.

10. The method of operating a propane dewaxing system which includes heat exchangers operated under high pressure, a warm solution tank and a plurality of chillers, which method comprises cooling a propane waxy-oil solution in said heat exchangers to a temperature which is slightly above the visual cloud point of said solution, flash chilling said solution from said temperature to a temperature just below the actual cloud point by maintaining the warm solution drum at a pressure in the range of about 80 to 125 pounds per square inch gauge which will permit such flash chilling and introducing said flash chilled solution into a chiller without raising the temperature of said solution substantially above the actual cloud point thereof.

11. The method of claim 10 which includes the step of accumulating flash chilled solution in said solution drum while maintaining said drum at a pressure sufficiently low to prevent the temperature of said solution from exceeding the actual cloud point temperature of the solution.

12. The method of claim 10 which includes the step of removing flashed vapors along with liquids from the solution drum to the chiller at substantially the same rate as flash cooled solution and vapors are introduced into the solution drum and operating said solution drum with substantially no liquid level therein.

13. In a chilling system for a propane dewaxing plant which comprises a holding compressor with its associated inlet line and discharge line, a warm solution drum connected through pressure control valves with the compressor discharge line, a chiller connected by a solution inlet line to the solution drum, a heat exchanger connected by a valve-containing line to the solution drum, a chilling compressor discharging into the holding compressor discharge line and connections between the chiller and the chilling compressor, the improved structure which comprises a connecting line between said warm solution drum and said holding compressor inlet line and a pressure control valve in said connecting line for enabling the warm solution drum to operate at a substantially constant pressure sufficiently low to effect flash chilling of solution from slightly above its visual cloud point through a range of at least about 5° F. but not more than about 25° F. as it passes through the valve-containing line between said exchanger and said warm solution drum.

14. The method of increasing the filterability of a solid crystal from a propane solution of an oleaginous material, which method comprises heating a propane solution of oleaginous material containing a crystallizable solid to a temperature sufficiently high to effect complete solution of said oleaginous material and solid and under a pressure sufficiently high to maintain the propane in liquid phase during the heating step, cooling the heated solution while maintaining it under said high pressure to a temperature slightly above the visual cloud point of said solution, then shock chilling said solution by quickly passing it from a zone of said high pressure to a zone of low pressure, said zone of low pressure being at a pressure sufficient to maintain the solution at a temperature near but below the actual cloud point of the solution, then further chilling the shock chilled solution by evaporative cooling to effect the production of filterable crystals of the solid to be separated.

15. The method of claim 14 wherein the oleaginous material is a fatty material.

16. The method of claim 14 wherein the oleaginous material is a hydrocarbon.

17. The method of claim 14 wherein the flash chilling is through a temperature range of at least about 5° F. but not more than about 25° F.

KENNETH H. WANDERER.
LEONARD A. HAYS, JR.
ROBERT B. SELUND.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,974,398	Ellsberg	Sept. 18, 1934
2,143,872	Forrest et al.	Jan. 17, 1939
2,229,659	Carr	Jan. 28, 1941
2,260,994	Knowles	Oct. 28, 1941
2,265,139	Brandt	Dec. 9, 1941
2,287,966	Brandt	June 30, 1942
2,303,721	Brandt	Dec. 1, 1942