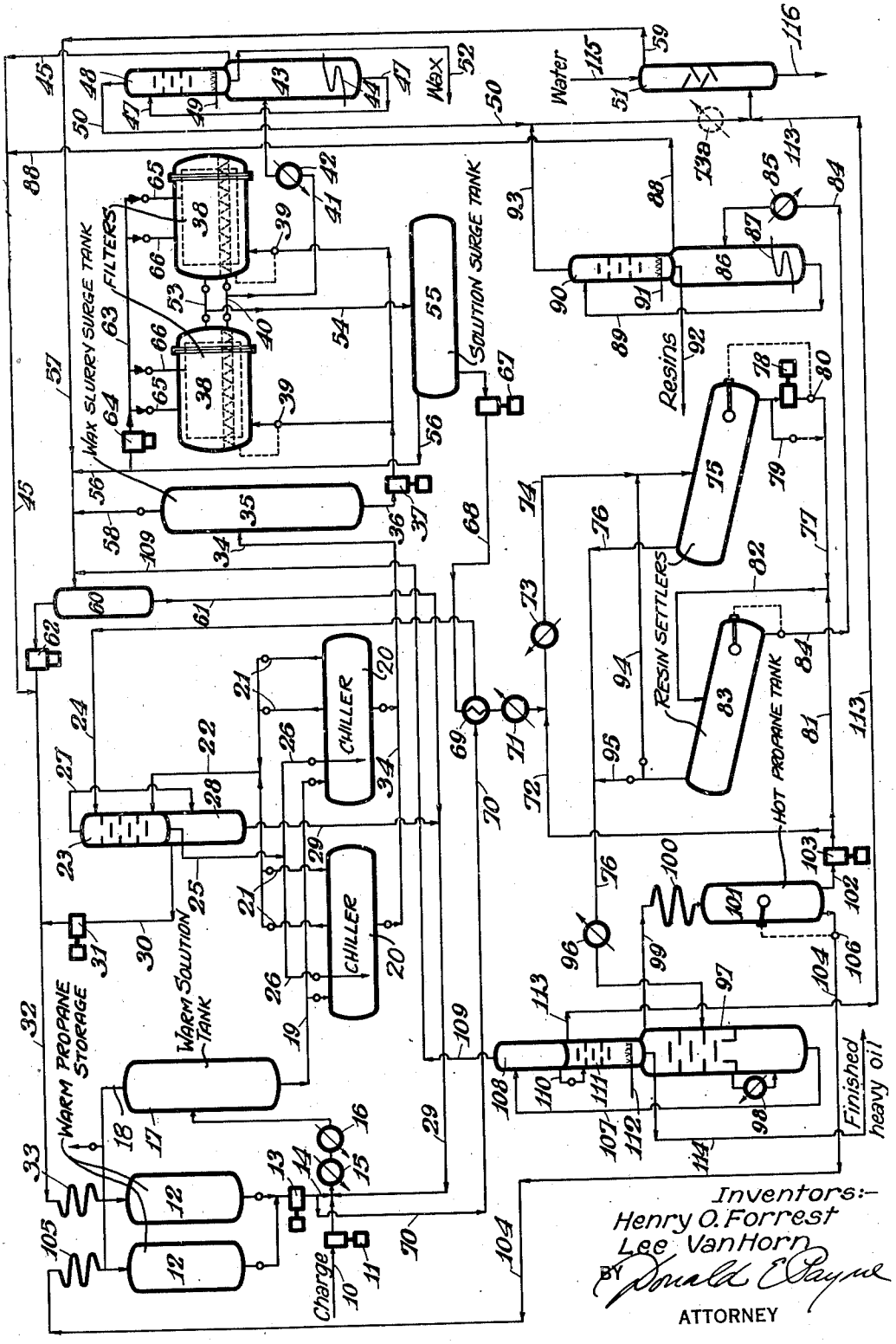
Jan. 17, 1939. H. O. FORREST ET AL 2,143,872

REFINING LUBRICATING OILS WITH PROPANE

Filed Dec. 31, 1936



Inventors:-
Henry O. Forrest
Lee VanHorn
BY Donald E. Payne
ATTORNEY

Patented Jan. 17, 1939

2,143,872

UNITED STATES PATENT OFFICE

2,143,872

REFINING LUBRICATING OILS WITH PROPANE

Henry O. Forrest, Teaneck, and Lee Van Horn, Westfield, N. J., assignors to Standard Oil Company, Chicago, Ill., a corporation of Indiana

Application December 31, 1936, Serial No. 118,536

5 Claims. (Cl. 196—13)

This invention relates to the refining of lubricating oils with propane, and it pertains more particularly to an improved apparatus and method for chilling in the dewaxing step and improved methods and apparatus for refining hydrocarbon oils containing resins. In certain propane dewaxing processes we have found that the vapor velocities in the chillers are so great as to cause an undue amount of foaming and entrainment when the chilling is effected in vertical towers. One object of our invention is to provide a new and improved system for chilling propane oil solutions by evaporative cooling in horizontal chillers without losing the advantages that we have heretofore obtained by introducing cold propane onto bubble plates in the upper part of the vertical chillers. In other words, our object is to provide a method and means for utilizing the refrigeration value of cold propane and to simultaneously avoid entrainment and prevent shock chilling by contacting propane vapors leaving the horizontal chiller with refrigerated propane prior to the introduction of the refrigerated propane into said chillers for "make-up" diluent and refrigerant.

A further object is to provide in an improved system a new combination of propane refining processes particularly adapted for the treatment of Pennsylvania cylinder stocks or other viscous lubricants which are substantially free from asphalt but which contain waxes and resins. A further object is to provide an improved combination of dewaxing and deresinating steps with an improved propane recovery system which will be more simple and economical than systems heretofore employed. A further object is to provide optimum operating conditions for the refining of Pennsylvania cylinder stocks and similar lubricating oil fractions.

In practicing our invention we first heat the stock with propane to a temperature far above that required for complete wax solution, and we then chill the mixture, employing a plurality of horizontal batch chillers. By employing horizontal chillers we obtain lower vapor velocities and less foaming and entrainment than we would have in vertical chillers of the same diameter, and we can remove the propane at spaced points along the chiller at rates which effect optimum wax crystallization. To cooperate with the horizontal chillers we provide a combination gas washer and trap. The propane from the chillers is scrubbed with cold propane in the washer, thus warming up the cold propane so that it will not cause shock chilling when added to the oil-propane mixture in the chillers. Propane leaving this scrubber is passed through a trap and thence to the compressor, liquids from the trap being returned for admixture with oil and propane prior to the heating step.

We have discovered that for Pennsylvania cylinder stocks or similar oils the optimum feed mix ratio of propane to oil is about from 2:1 to 2½:1. The warm mix ratio in the chiller is about 2½:1. The cold mix ratio in the chiller is about 3:1. The filtering ratio is about 3:1 and the deresinating ratio is about 10:1. Although it is inherent in a propane dewaxing process that certain resins or color bodies are removed from the oils with the wax, we have found that it is desirable to include a high temperature deresinating treatment after the dewaxing step, preferably effecting a fractionation at a temperature of about 180° F. followed by a fractionation or washing of the resins at a temperature of about 150° F. We have found that considerable economies can be effected by increasing the temperatures and pressures which have heretofore been employed in depropanizing the finished oil so that this propane may be liquefied and stored at about 130° F. for use in the deresinating step, this hot propane storage system being operatively connected with the warm propane storage system heretofore employed in dewaxing processes.

Our invention will be more readily understood from the following detailed description of a preferred embodiment and from the accompanying drawing which forms a part of this specification, and which diagrammatically represents a plan partly in elevation, of our improved system.

We will describe the invention as applied to the refining of a Pennsylvania cylinder stock having a viscosity of about 145 seconds Saybolt at 210 F. and we will describe the operating conditions, quantities and proportions for a plant designed to handle about 1100 barrels/day of such charging stock. It should be understood, however, that our invention is applicable to the refining of any oil stocks from any crude source, provided the necessary adjustments are made in proportions and operating conditions, as will be apparent to those skilled in the art. While we refer to propane as our refining agent, it should be understood that the propane may contain or be replaced by other normally gaseous hydrocarbons, particularly ethane, isobutane and butane, propylene, butylene, etc. We prefer to employ a good grade of commercial propane.

About 14,440 pounds/hour of charging stock is introduced through line 10 and pump 11 for ad-

mixture with 19,200 pounds/hour of propane from storage tanks 12, pump 13 and line 14. This mixture, which is about 2.35:1 by volume, is passed through heater 15 which raises the temperature of the mixture to about 190° F. We have found that the filterability of the wax can be markedly improved by this initial heating to a temperature which is far above that required for complete wax solution. Although temperatures as low as 140 to 150° F. may be satisfactory for some stocks, in the present case we advise heating to 190° F. Having superheated the mixture to insure proper wax crystallization, we next chill it in cooler 16 to a temperature of about 115° F. by heat exchange with cooling water and then to a temperature of about 70° F. to 100° F. by heat exchange with the dewaxed solution which has previously been used to chill propane, as will hereinafter be described; (to avoid complexity this last heat exchange is not shown in the drawing). The mixture is then introduced into warm solution tank 17 which is designed to operate at 225 pounds gauge with a warm solution at 100° F. which is preferably maintained in equilibrium with propane storage tanks 12 through line 18.

Warm solution from tank 17 is withdrawn through line 19 into one of the horizontal chillers 20, the chiller having previously been conditioned to avoid shock chilling on the introduction of solution. Chilling is effected batchwise by removing propane vapors from the chiller through line 21 and line 22 to the base of scrubber 23, it being understood, of course, that one chiller is being cooled while another is being emptied, as many chillers as necessary being employed for maintaining continuous operation of the filters.

Cold propane is introduced through line 24 at the top of scrubber 23 and is contacted with the warm propane vapors therein. The condensation of the propane in this scrubber warms up the cold propane to a sufficient extent to permit its introduction through lines 25 and 26 into chillers 20 for maintaining the desired amount of propane in the chillers at all times, and regulating the cooling rate. Uncondensed propane from the top of scrubber 23 is introduced through line 27 into trap 28 wherein liquids are separated out, and returned through line 29 to be admixed with incoming propane and oil stock. Vapors from trap 28 are led through line 30 and compressor 31 to line 32 and condenser 33, wherein the vapors are liquefied and from which they are returned to propane storage tanks 12.

Slurry from the base of chillers 20 is introduced through line 34 to wax slurry surge tank 35, preferably at a temperature of about —45° F. and with a propane ratio of about 3:1 by volume (14,440 lbs./hour of stock and 24,560 lbs./hour propane). The amount of propane introduced through line 24 in our system is 21,045 lbs./hour, but this amount will necessarily vary with the effectiveness of heat exchange facilities, insulation, etc.

Slurry is introduced through line 36 by pump 37 to filters 38, the amount of slurry introduced into each filter being controlled by liquid level control valves 39. The filters are preferably of the type disclosed in Keith et al. U. S. Patent 2,050,007.

Wax cake from the filters is withdrawn by screw conveyors and pumps through lines 40 and 41 and through heater 42 which heats the mixture to about 180° F., at which temperature it is introduced into still 43 which is maintained at about 215 pounds gauge and with a bottom temperature of about 260° F. by steam coils or reboiler 44. Propane from the top of still 43 is withdrawn through line 45 to line 32. Hot solution from the bottom of still 43 is introduced through line 47 into stripper 48 where it is stripped with steam from line 49, the steam and propane being withdrawn through line 50 to condenser 51. With the 1970 pounds/hour of wax (petrolatum), there are 2870 pounds/hour of propane thus introduced into the recovery system, of which propane about 2745 pounds/hour are withdrawn through line 45 and about 125 pounds/hour through line 50. The wax is withdrawn through line 52.

Returning now to the filter system, filtrate is removed through lines 53 and 54 to dewaxed solution surge tank 55 the vapor space of which is connected by line 56 to low pressure gas line 57. The wax slurry surge tank 35 is also connected by line 58, and condenser 51 is connected by line 59, to this same gas line 57 which conducts the gases to trap 60 from which liquids are conducted through line 61 to line 29 and gases are compressed by compressor 62 discharging into line 32.

Blow-back and pressuring gas may be supplied to the filter system through line 63, compressor 64 and lines 65 and 66. The filters are also preferably provided with connections for introducing cold wash propane, the cold propane lines being vented to line 57, or to line 63.

Dewaxed solution from tank 55 is passed by pump 67 through line 68 and heat exchanger 69, wherein it is warmed by propane from storage tanks 12 and line 70. It is this cooled propane from line 70 which is introduced through line 24 at the top of scrubber 23 as hereinabove described.

The dewaxed oil solution, about 12,470 pounds of oil and 24,800 pounds of propane per hour is heated in this exchanger from —40 to about +40° F. and is then further heated, preferably by suitable exchangers 71 to a temperature of about 145° F. at which temperature it is admixed with about 45,600 pounds/hour of propane in line 72 at about 130° F. The combined solution is heated from 135° F. to about 180° F. in heater 73 and at this temperature is introduced through line 74 into heavy oil settler 75 and is maintained under a pressure of about 570 pounds gauge. Thus we have in this settler about 10 volumes of propane per volume of heavy oil, and at the temperature of 180° F. we find that about 11,180 pounds/hour of deresinated oil and 96,000 pounds/hour of propane are withdrawn therefrom through line 76 (including recycled or recovered oil, as hereinafter described). The precipitated resins are withdrawn from the base of settler 75 through line 77 by means of a pump 78 which can be rendered ineffective by line 79 when liquid level controlled valve 80 is closed.

To 2530 pounds/hour of primary resins and 2800 pounds/hour of propane in line 77 we admix about 21,100 pounds/hour of propane from line 81, the warm mixture being introduced through line 82 into resin settler 83 which is maintained at about 150° F. and 570 pounds gauge. Resins are withdrawn through line 84 to heater 85 which raises the temperature of the 1290 pounds of resins and 1400 pounds of propane to 320° F., at which temperature the mixture is introduced into flash tower 86 operating at 215 pounds gauge and having a bottom tem-

perature of 320° F. maintained by reboiler 87. The propane from tower 86 is returned through line 88 to lines 45 and 32 about 1350 pounds/hour. Resins from the bottom of tower 86 are introduced through line 89 into stripper 90 into which steam is introduced through line 91, finished resins being withdrawn through line 92. Propane and steam from the top of line 89 is introduced through line 93 into line 50.

The propane-oil solution which separates from the resins in separator 83 may be returned to line 74 through line 94, or it may be directly introduced to line 76 through line 95. The solution in line 76 is heated by heater 96, which may be a plurality of exchangers, to a temperature of about 160° F., at which temperature it is introduced into still 97 which is maintained at a pressure of 295 pounds gauge with a bottom temperature of 270° F. which is maintained by re-boiler 98. Propane leaving the upper part of flash tower 97 through line 99 is liquefied in condenser 100 at a temperature of about 130° F., and is stored in hot propane surge tank 101, this tank being designed to operate up to pressures of 320 pounds gauge. Hot propane from this tank is withdrawn through line 102 and pump 103 to lines 72 and 81 for use in the fractionation processes in settlers 75 and 83. Propane from tank 101 may also be passed through line 104 and condenser 105 to propane storage tanks 12, the amount of propane being regulated by liquid level controlled valve 106.

Hot oil from the base of tower 97 is introduced through line 107 to low pressure flash tower 108 from which propane is led by line 109 to line 57. This flash tower 108 is preferably operated at about 260° F. and about five pounds gauge. The oil is then passed through line 110 to stripper 111 where it is stripped with steam from line 112, the steam and propane being passed through line 113 to line 50. The finished heavy oil is withdrawn from the base of the stripper through line 114 at a temperature of about 255° F. In the above system about 6850 pounds/hour propane is passed through line 99 to the hot surge tank, about 900 pounds/hour of propane vapor is passed through line 109 to line 57 and about 50 pounds/hour of propane vapor is passed through line 113 together with steam to the condenser 51.

Using about 750 pounds/hour of steam in stripper 111, 135 pounds/hour in stripper 48 and 85 pounds/hours in stripper 90 we find it advisable to employ a stripping steam condenser as a heat exchanger to furnish some of the heat for warming up the propane-oil mixture entering the deresinating step, as indicated in dotted lines by exchanger 73*a*. By this expedient we may condense most of the steam so that only about fifteen gallons per minute of water will have to be introduced through line 115 to condense the remaining steam and remove it through line 116 to the sewer.

While we have described in detail the preferred example of our invention, it should be understood that certain details have been omitted because of the already complex flow diagram, but with the proportions and conditions set forth one skilled in the art can readily practice the invention. We do not limit ourselves to the particular details hereinabove described except as defined by the following claims which should be construed as broadly as the prior art will permit.

We claim:

1. In a propane refining system for dewaxing and deresinating, a warm propane storage tank, means for admixing propane therefrom with waxy oil and for dewaxing said waxy oil in propane solution, means for recovering propane from the separated wax and returning said propane to said storage tank, a hot propane surge tank, means for mixing hot propane from said surge tank with dewaxed oil solution, means for deresinating said hot solution at an elevated temperature and pressure, means for recovering propane from said separated resins, means for depropanizing the hot oil solution at a temperature of about 270° F. and a pressure of about 300 pounds gauge, means for condensing the high pressure propane from said depropanizing means and introducing it into said hot propane surge tank at a temperature of about 130° F., means for passing propane from said hot propane surge tank to said propane storage tank and means for further recovering propane from oil discharged from the depropanizer and for returning said recovered propane to said propane storage tank.

2. A process for refining a wax-bearing oil with propane which comprises dissolving said wax-bearing oil in propane, introducing said solution into an elongated horizontal chilling zone, removing vapors from the upper part of said chilling zone, countercurrently contacting said removed vapors with cold propane whereby some of the vapors are condensed and any entrained oil is recovered in the cold propane, introducing the cold propane after the scrubbing step into the chilling zone, passing propane vapors from the scrubbing step into a trap-out zone, compressing and condensing propane vapors from the trap-out zone and returning the condensed vapors to the solution step, collecting liquids in the trap-out zone, and separately returning said liquids for admixture with the propane-waxy oil solution.

3. The method of refining substantially asphalt-free-cylinder stock which comprises maintaining a supply of warm propane, mixing said stock with about 2½ to 3 volumes of warm propane, from said supply heating the mixture to about 150 to 190° F., cooling the heated mixture to effect the crystallization of wax in separable form, separating the wax from the propane solution at a low temperature, increasing the propane ratio of the dewaxed oil solution to about 10 volumes of propane per volume of dewaxed oil, heating the mixture to about 150 to 180° F. at a pressure sufficient to maintain the propane in a liquid state for effecting phase separation of two liquid phases, removing a portion of the propane from one of said phases at a pressure of the order of 300 pounds per square inch, condensing the propane from said propane removal step at a temperature of the order of 130° F., maintaining a supply of this hot propane and using this hot condensed propane for increasing the propane ratio of the dewaxed oil subsequent to the dewaxing step and prior to the high temperature separation step.

4. In apparatus of the class described, a warm propane storage tank, a chill chamber, means for mixing propane from the storage tank with a waxy oil and for introducing said waxy oil propane mixture into said chill chamber, a scrubbing chamber, a source of cold propane, means for passing gases from the chill chamber and cold propane from said source, respectively, to said scrubbing chamber, means for returning liquid from said scrubbing chamber to said chiller, a filter, means for transferring chilled propane-oil-wax slurry from said chillers to said filter, means for separately removing oil and wax from said

filter, a hot propane storage tank, means for adding hot propane from said hot propane storage tank to the means for removing oil from said filter to form a hot mixture of filtrate and propane, means for heating the hot mixture to a temperature of about 150 to 180° F., means for separating two liquid phases from the heated mixture, systems for separately depropanizing said separate liquid phases, means for condensing propane from one of said depropanizing systems at high temperature, and means for returning said high temperature propane condensate to said hot propane storage tank.

5. The apparatus of claim 4 which includes means for passing propane from the warm propane storage tank through a cold filtrate exchanger to provide the source of cold propane and to warm up the cold filtrate.

HENRY O. FORREST.
LEE VAN HORN.