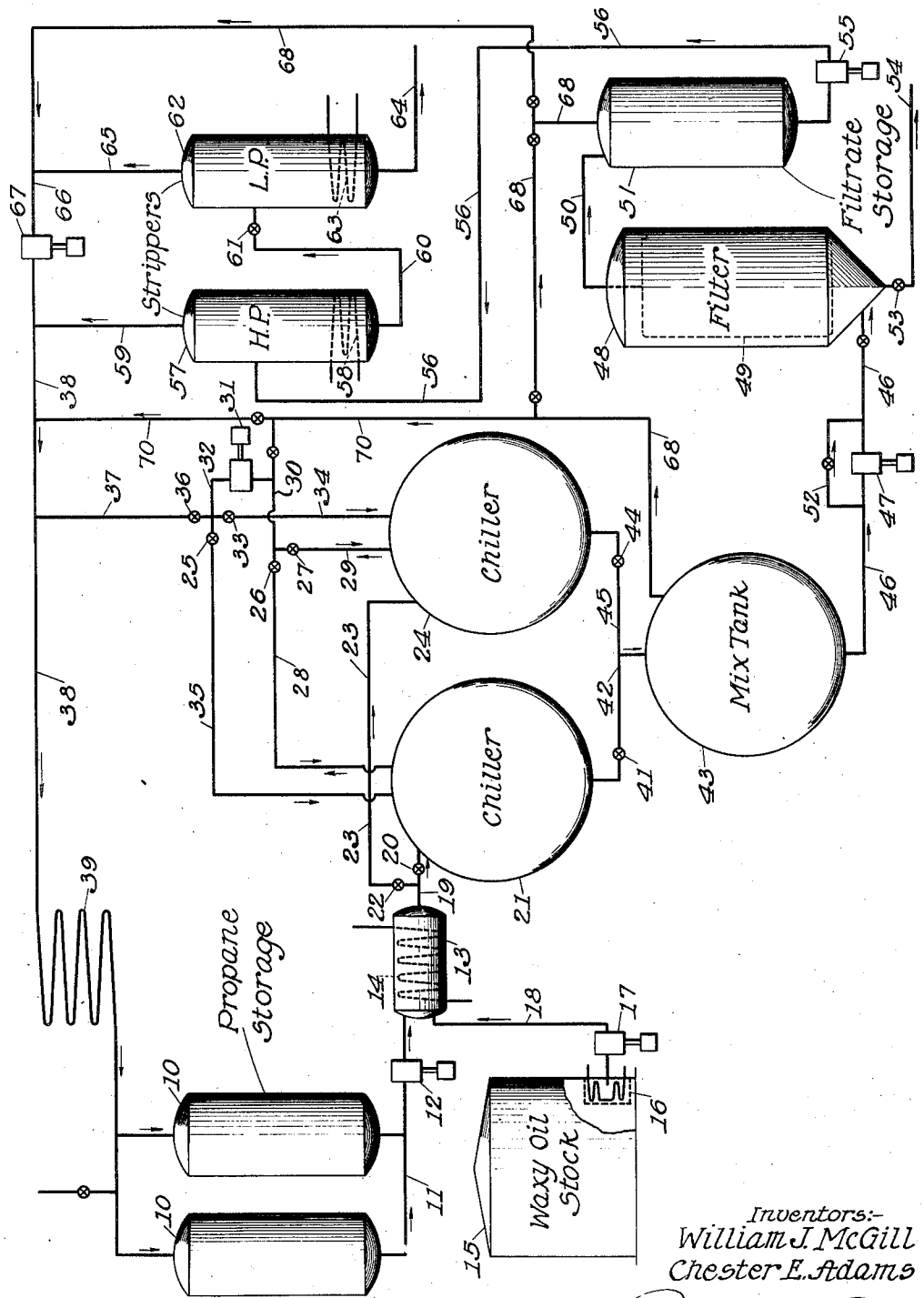
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PROPANE DEWAXING PROCESS

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PROPANE DEWAXING PROCESS

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This invention relates to a dewaxing system and it pertains more particularly to a system for the separation of wax from petroleum lubricating oils by means of liquefied normally gaseous diluent-refrigerants such as propane. When oil is diluted with liquid solvents and chilled by means of brine coils in accordance with conventional practice, the temperature must be lowered very slowly, only a few degrees an hour; otherwise the temperature differential between the diluted oil and the brine coil will be too great and there will be shock chilling at the surface of the brine coil. When the chilling is effected by direct vaporization of propane from a propane lubricating oil stock mixture, the cooling may be about fifty or sixty times as fast as was possible with brine coolers,—it may be from about one-half degree to eight degrees per minute, preferably about three degrees per minute. However, when a solution of waxy oil in propane hits a cold metal surface or a cold body of liquid, there is an instantaneous cooling of the mixture at a theoretically infinite rate. In other words, for an extremely small fraction of a second the cooling rate may be several hundred degrees per minute. This results in shock chilling and it causes the formation of wax crystals which are so fine that they cannot be readily removed from diluted oil by filtration and/or other mechanical means. The object of our invention is to prevent this shock chilling in a propane dewaxing system.

A further object of our invention is to provide means for cooling a solution of wax and oil in propane so that wax will crystallize out in readily separable form, so that the yield of finished oil will be thereby increased, so that the finished oil will have a low pour point, and so that the mechanical separation of wax from diluted oil will be expedited. A further object is to minimize compressor costs, to utilize more effectively the refrigeration which is available in the system, to effect a saving of steam and condenser water, and to increase the efficiency of the dewaxing process generally.

In practicing our invention we use a plurality of chill chambers seriatim; propane removed from one chill chamber to effect refrigeration is condensed in the next chill chamber to warm it up. When the temperature in the two chambers approaches equilibrium and the cooling rate slows up, we continue the removal of propane vapors from the first chill chamber by means of a compressor and we force the compressed propane into the second chill chamber. In this way the temperature of the first chamber may be reduced to —35 or —40° F., while the temperature of the second chamber is raised to about +90° F. or it is raised to the temperature of the incoming solution of wax and oil in propane. The second chamber is then charged with warm solution and the warm solution is chilled by removing propane vapors and condensing them in the first chamber. When the rate of cooling falls off the compressor is again used, this time to remove vapors from the second chamber and to force them back into the first chamber. In this way it will be observed that, to a large extent, the refrigerant is merely crossed over from one chill chamber to the other, and that the cold slurry leaving a chill chamber is augmented by a considerable amount of propane condensed in the empty chamber before charging.

The invention will be more clearly understood from the following description and from the accompanying drawing wherein we have illustrated diagrammatically the preferred embodiment of our improved dewaxing system.

In describing the invention we will refer to the separation of wax from a heavy lubricating distillate stock from midcontinent crude, the distillate having been acid treated and neutralized, and having a viscosity of about 90 seconds Saybolt at 210° F. It should be understood, however, that the invention is equally applicable to the separation of wax from any overhead or residual lubricating stock from any wax-bearing crude, treated or untreated.

Propane that is used in this specification is illustrative only of normally gaseous substances which may be liquefied to serve as diluent-refrigerants in this type of a dewaxing process. We prefer to use ordinary commercial propane which may contain a fraction of a percent of ethane and 8% or 10% of isobutane and/cr normal butane. The propane may contain larger amounts of butane, and it may contain unsaturates such as ethylene and propylene. Also, we may use liquefied normally gaseous substituted hydrocarbons, such as methyl chloride, dichlor difluor methane, etc., or we may use normally gaseous ethers such as methyl ether or amines such as dimethylamine. The word "propane" as used throughout this specification should not be limited to that particular compound but should be understood broadly to include the equivalents thereof. In other words, propane will be considered to mean a liquefied normally gaseous

diluent-refrigerant compound or mixtures of compounds.

Referring to the drawing, propane in storage tanks 10 may be transferred through pipes 11 by means of pump 12 to mixer 13, which may be provided with steam coil 14 for effecting complete solution of both oil and wax in the propane. The waxy oil stock from storage tank 15 is withdrawn through discharge heater 16 and forced by pump 17 through pipe 18 to mixer 13. From the mixer the solution of oil and wax in propane is discharged through pipe 19 and valve 20 into horizontal chamber 21 (valve 22 being closed), or through valve 22 and pipe 23 to chill chamber 24 (valve 20 being closed).

We will assume that chamber 24 has just been emptied and is at a temperature of about —40° F. and that the incoming warm solution is introduced into chamber 21 which is at a temperature of about +90° F. Valve 25 is closed and valves 26 and 27 are opened so that propane vapors from chamber 21 may pass through pipe 28 and pipe 29 to the cold chamber 24. This chamber contains a large mass of iron, and thus it acts as a storage medium for conserving refrigeration. Gradually, in about ten or fifteen minutes, the temperature in chamber 21 has fallen and the temperature of chamber 24 has risen until substantial equilibrium has been reached. To continue the cooling we close valve 27 and withdraw propane vapors through pipe 30 by means of compressor 31, which forces these vapors through pipe 32, open valve 33, and pipe 34 into chamber 24. Compressor 31 is of the variable type and is designed to carry the pressure from 70 to 100 pounds down to zero pounds gauge on the suction side and to compress the gases to about 150–160 pounds/square inch. As these condensed gases are forced into chamber 24 they are condensed under pressure and they thereby heat the chamber to the desired point, which is usually the temperature of the incoming stock, about 90° F. At this point, additional compressed gases may be by-passed through valve 36, pipe 37, pipe 38, condenser 39 and pipes 40 back to propane storage tanks 10.

As soon as the temperature in chamber 21 reaches —40° F., valve 41 is opened and the slurry is allowed to pass through pipe 42 to mix tank 43. Valve 44 and pipe 45 serve to convey chilled slurry in the same way from chiller 24 to the mix tank.

When the slurry has been transferred from chiller 21 to the mix tank, valves 41 and 44 are closed and a warm solution of oil and wax in propane is introduced into chiller 24 which has been heated to the temperature of the incoming solution by condensation of compressed propane. The mixture in this chiller is then cooled by allowing vapors to pass directly through pipe 29, valves 27 and 26, and pipe 28 to chiller 21. When equilibrium is reached, valve 26 is closed and propane is withdrawn through pipe 29 by compressor 31 and forced through pipe 32, valve 25, and pipe 35 to chiller 21, valves 33 and 36 being closed. In this manner chiller 21 is again warmed up preparatory to receiving another batch of warm propane oil-wax solution.

The mix tank acts as a supply or storage for chilled slurry which is forced through pipe 46 by means of pump 47 to closed filter 48. The diluted oil passes through the filter leaf 49 and is withdrawn through pipe 50 to filtrate storage 51. When a sufficient wax cake has been built up, the excess slurry in the filter is forced back through pipe 46 and by-pass 52 to the mix tank 43, valve 53 is opened and the wax slurry is removed through a screw conveyor or the line 54 to a suitable stripping means for removing propane from the wax. If desired, we may wash the wax cake with fresh propane. For the sake of simplicity, this feature is not shown in the drawing.

The filtrate is forced from storage 51 by pump 55 through pipe 56 to high pressure stripper 57, which is heated by steam coil 58. The propane is conveyed by pipe 59 to high pressure propane line 38. The hot oil is conveyed through pipe 60 and reducing valve 61 to low pressure stripper 62, which may also be heated by steam coil 63 and which may be provided with suitable stripping means. Dewaxed oil is removed through pipe 64 and the last traces of propane are removed through pipe 65 to low pressure gas line 66 which leads to the suction side of compressor 67. This compressor boosts the pressure of the propane up to about 150–160 pounds and it discharges into pipe 38.

Mix tank 43, filtrate collector 51 and other vessels which must be maintained at about —40° F. are connected by pipes 68 to the suction side of compressor 67. This compressor may also be manifolded with compressor 31 by valved line 70.

The operation of our invention will be apparent from the description of the apparatus. A large part of the propane which serves as refrigerant is already in the chill chamber when the solution of oil and wax in diluent propane is added to the chill chamber. The refrigerant-propane is transferred to another chill chamber partly by vaporization due to the initial difference in pressures and partly by means of compressor 31, so that as the first chill chamber is cooled, the second chill chamber is heated. The condensation of propane vapors in the second chamber serves to dissolve all traces of wax from the walls thereof, and prevent contamination of the ensuing charge by wax from a previous run. Shock chilling is entirely avoided. The transfer of the propane oil-wax mixture through a plurality of cooling stages is avoided. The use of steam for warming up the chill chamber is avoided. The amount of required condenser water is markedly decreased and the use of a compressor for removing high pressure propane vapors from the chill chamber is avoided and a minimum degradation of wax crystal structure is attained. It will thus be seen that our improved system effects great economy in compressor costs, steam consumption and condenser water requirements. The temperatures referred to in this specification are all expressed in degrees Fahrenheit.

While we have described the use of two chill chambers, it should be understood that we may use three or even more. For instance, one chamber may be filled while another is being chilled, a third is being emptied and a fourth is being warmed up. When a larger number of chillers is used, we may transfer the vapors to cold chambers in rotation, always condensing the warmest vapors in the warmest chambers. Various other modifications will be apparent to those skilled in the art and we do not limit ourselves to any of the details hereinabove set forth except as defined by the following claims, which should be construed as broadly as the prior art will permit.

We claim:

1. The method of operating a propane-dewaxing system which comprises compressing propane into a chilling zone to raise the temperature of the zone to that at which wax and oil are soluble in propane, introducing a solution of oil and wax in propane into the chilling zone whereby it will be combined with the liquefied propane therein, and cooling the solution to effect solidification of wax in readily separable form by vaporization of propane from said chilling zone.

2. The method of claim 1 wherein the propane vapors removed from said chilling zone are condensed in another chilling zone to raise the temperature thereof to that at which wax and oil are soluble in propane.

3. The method of operating a propane dewaxing system which comprises compressing propane into a chilling zone to raise the temperature of said zone sufficiently high so that incoming propane-oil-wax mixture will not be shock chilled on contact therewith, introducing an oil-wax-propane mixture into said chilling zone so that it will be combined with the liquefied propane therein, cooling the mixture to effect solidification of wax in separable form, removing solidified wax from the propane oil solution, and removing propane from the dewaxed oil.

4. The method of claim 3, which includes the steps of maintaining a large volume of said chilled propane-oil-wax mixture and introducing said chilled mixture from said large body into an external wax removal zone while maintaining the mixture at a sufficiently low temperature to prevent substantial solution or melting of the wax and preventing the mixture from coming in contact with a cold body to cause shock chilling.

5. The method of preventing shock chilling in the process of separating wax from a diluted oil-wax mixture by means of a liquefied normally gaseous diluent-refrigerant such as propane, which comprises bringing the temperature of the chilling zone to substantially the same temperature as that of the diluted oil-wax mixture prior to the introduction of the mixture into the chilling zone whereby the diluted oil-wax mixture will not come in contact with cold surfaces.

6. The process of claim 5 wherein a cool chilling zone is brought to the same temperature as a warmer diluted oil-wax mixture by condensing propane in said cooled chilling zone, the propane remaining therein to serve as additional diluent-refrigerant.

7. In a propane dewaxing system, the method of warming a chiller prior to the introduction of a diluted oil-wax mixture thereinto, which comprises introducing propane vapors into said chiller under such pressure that said vapors will condense in said chiller and thereby raise its temperature to that of the incoming diluted oil-wax mixture.

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