June 28, 1938. C. E. ADAMS ET AL 2,122,019

SEPARATION OF WAX FROM OILS

Filed Dec. 1, 1934 2 Sheets-Sheet 1

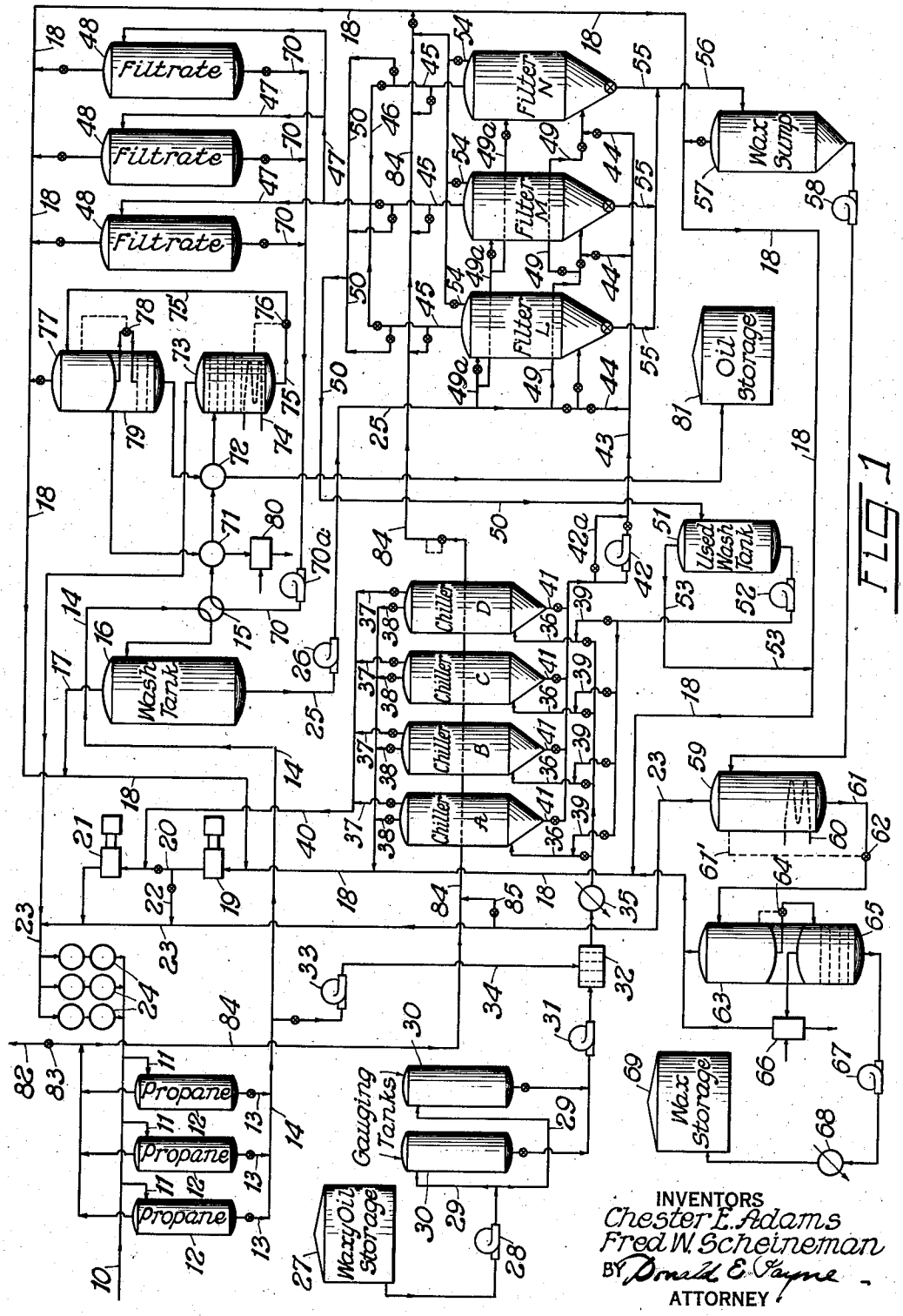


FIG. 1

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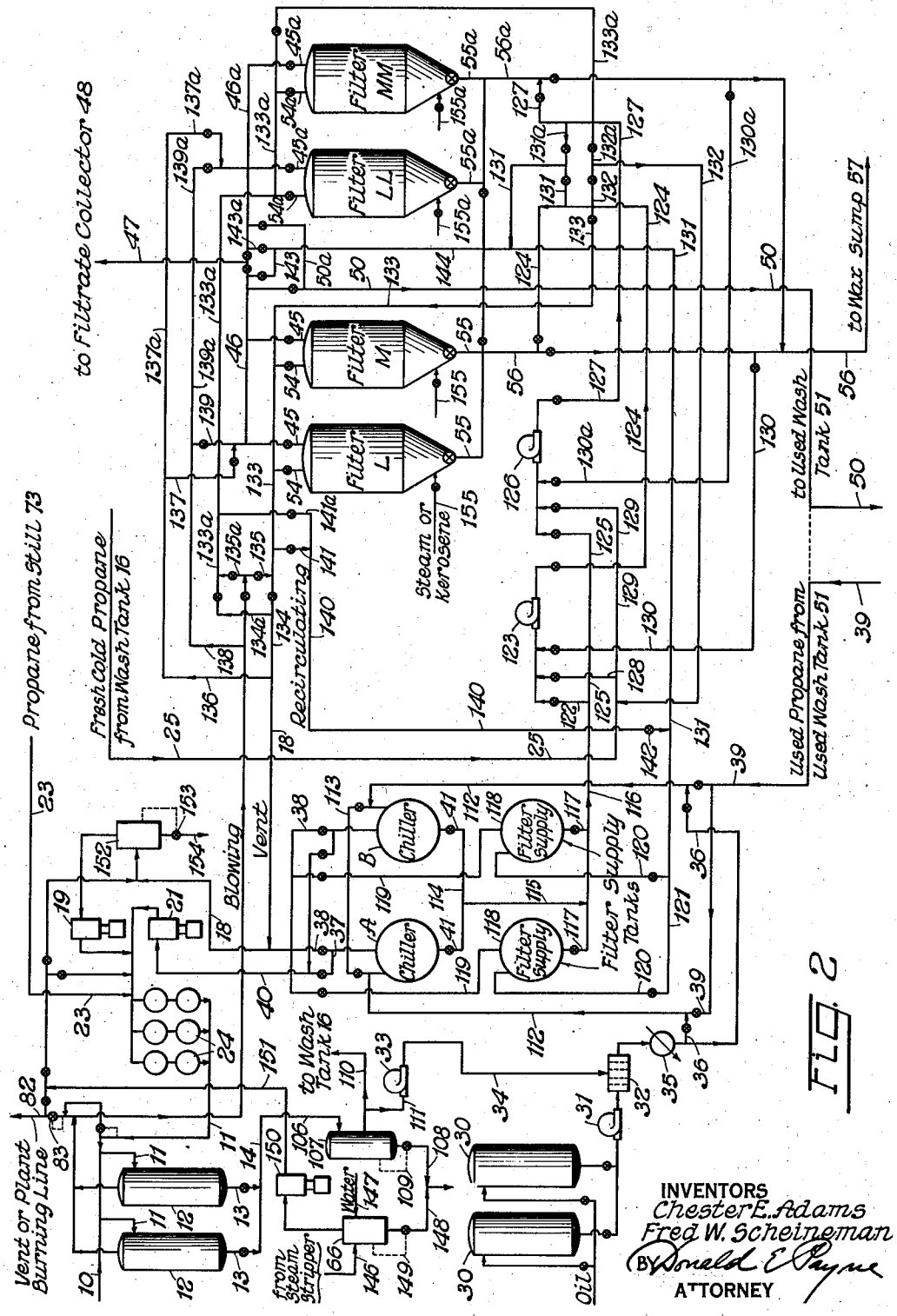


FIG. 2

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2,122,019

SEPARATION OF WAX FROM OILS

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Application December 1, 1934, Serial No. 755,602

8 Claims. (Cl. 196—18)

This invention relates to the separation of wax from oils and it pertains more particularly to an improved system for the use of normally gaseous diluent refrigerants, such as liquid propane. This application is a continuation in part of applicants' copending application No. 599,730, filed March 18, 1932.

The object of our invention is to provide a commercial large-scale dewaxing plant for petroleum refineries in which inflammable volatile diluents or refrigerants may be used with safety. In other words, our object is to provide a closed system and means for regulating the pressure in all parts of the system so that leakage of propane will be prevented. We want to avoid the necessity for opening filters and this means, in turn, that we must provide a system whereby the filter leaves will not become clogged with amorphous or finely divided wax. Our object is to provide a special design for filters that will fit into a specially designed filter system to accomplish the continuous dewaxing of oil in a closed system which will not have to be frequently interrupted for repair or cleaning.

A further object of our invention is to provide an improved system and method of chilling diluted oil-wax mixtures so that the optimum wax crystal formation may be obtained. In other words, we want to determine the sequence of steps, the relative volumes of diluent refrigerant and waxy oil in the several steps, the optimum manner and rate of cooling, and the method of handling the crystals which will prevent them from being macerated or deformed prior to filtration. The particular method of crystal formation and the method of handling these crystals is of great importance in a commercial dewaxing system.

A further object of our invention is to provide a system for obtaining maximum mechanical, thermal, and operating efficiency. We want to arrange heat exchangers and chillers so that it will be possible to abstract all of the heat from wax-bearing oil without the use of the conventional wax chillers wherein scrapers are necessary for removing wax from cold walls. We also want to obtain the maximum benefit from the low temperatures of dewaxed oil and diluent so that the expense of refrigeration may be reduced to a minimum.

A further object is to provide improved means for eliminating water from all parts of the system.

A further object is to provide an improved method and means for chilling the oil-wax mixture to obtain readily filterable wax crystals, for effectively utilizing available refrigeration and "used propane" in this chilling step, and for providing improved methods and means of handling the slurry during and subsequent to the chilling step.

A further object is to provide an improved method and means for operating a wax filtration system whereby maximum flexibility of operation may be obtained by the use of a minimum number of pumps, conduits and connections. A further object is to provide improved means for washing wax cake, discharging the washed cake from the leaves, and utilizing the used wash propane in the system.

A further object is to obtain improved lubricating oils having high flash points and low pour points, and to obtain wax which is substantially oil free. Other objects will be apparent as the detailed description of our invention proceeds.

Certain features of our invention may be briefly characterized as follows: In the first place, we employ a dewaxing system which is tied together by a gas line, all parts of said system being operatively connected with said line. Vaporized propane from all parts of the system are sucked from this line by suitable pumps, compressed, condensed and returned to storage for re-use. This tying together of the whole system by suction gas lines insures safety, and at the same time it enables the temperature of each respective element of the combination to be separately controlled in a very simple and expeditious manner. In other words, the tying together of the whole dewaxing system with a suction gas line makes it possible to employ volatile and expensive refrigerants and diluents which were heretofore too dangerous for commercial use and makes it possible to improve the operation of filters, chillers, etc. so that the operation is uninterrupted over a long period of time.

Another feature of our invention is the combination with our suction gas line of an improved, closed vertical filter in which reverse flow and pressure regulation co-act to remove the filter cake and clean the filter cloth. Another feature of our invention is the combination of "wash propane" and "used wash propane" tanks with the chillers and filters so that practically all of the oil can be removed from the wax without the remelting or redissolving the same in oil or diluent. Another feature is our improved heat exchange system whereby cold oil-diluent mixtures are used to cool the waxy oil without the formation of wax crystals on the sides of containers so that the use of scrapers is avoided.

Another feature of our invention is the use of liquid traps for removing water from the system and for preventing liquids from entering the compressors. A further feature is the provision of safety means to adequately relieve the system of propane gas in case of failure of condenser, water or compressors.

Another feature of our invention is the improved system of chillers, filter supply tanks and connections from said chillers and supply tanks to the other elements of the system.

Another feature of our invention is the use of two banks of filters with separate charging means therefor and with means for obtaining maximum flexibility of operation with a minimum number of connecting lines. Another feature is the use of the wax discharge means as a means for introducing wax slurry into the filter.

A most important feature of our invention is the method of operating the chilling and the filtering system.

The invention will be more clearly understood from the following description taken in connection with the accompanying drawings which form a part of the specification and in which similar parts are designated by like reference characters throughout the several views. In these drawings:

Figure 1 is a diagrammatic plan of our entire system.

Figure 2 is a modification of the system illustrated in Figure 1 showing additional liquid trap-out systems and improved chilling and filtering units.

Our invention will be described as used for the separation of wax from a treated distillate lubricating oil from Mid-Continent crude petroleum, the particular distillate having a viscosity of about 100 seconds Saybolt at 210° F. and an A. P. I. gravity of 21.3. It should be understood, however, that the invention is equally applicable to any wax-bearing oils.

In the description of our preferred embodiment we will disclose the use of commercial propane as a combined diluent and refrigerant. It should be understood that other normally gaseous hydrocarbons, such as butane, isobutane, butylene, ethane, ethylene, propane, propylene, etc. may be used either separately or in admixture with each other. The waxy oil may be diluted all through the system with a small amount of filter naphtha or other light oil which may be useful as a viscosity breaker. Instead of using normally gaseous hydrocarbons we may use normally gaseous ethers such as methyl ether or we may use chlorinated or fluorinated hydrocarbons such as methyl chloride or dichlor-difluor-methane or oxygenated compounds such as ethylene oxide; the last named substances are useful in the dewaxing process because they have a tendency to throw wax out of solution. We may use normally liquid antisolvents for wax, such as acetone, acid esters, etc. or mixtures of liquid antisolvents with miscibility agents such as benzene or mixtures of benzene with naphtha. If liquids of this type are mixed with the oil they may be considered in the following description as a part of the oil. In our preferred embodiment we effect practically complete dewaxing of the oil by the use of commercial propane.

The propane enters the system through line 10 and is introduced by pipes 11 into storage tanks 12 which are designed to hold the propane at temperatures as high as 95° F. at which temperature the propane may exert a pressure of 165 pounds gauge (above atmospheric). The liquid propane may be selectively removed from one of these storage chambers through line 14, passed through heat exchanger 15, and thence into wash tank 16. The propane is usually at a temperature of 95° F. in line 14 and it is cooled in exchanger 15 to a temperature of about —20° F. The propane in wash tank 16 is cooled to a temperature of —40° F. by the evaporation of a part of the propane, vapors being removed from the tank through conduit 17 and line 18 to compressor 19 which is designed to maintain atmospheric pressure in line 18. The gases may then be passed through pipe 20 to compressor 21 which boosts them to a pressure of about 200 pounds, or compressor 19 may be designed to boost them to this pressure, in which case compressor 21 is cut out by pipe 22. We prefer to use compressor 19 as a holding compressor and compressor 21 as a chilling compressor, as will be hereinafter described. In any case, the highly compressed vapors are passed through line 23 and condensers 24 back to storage tanks 12. The cold propane from wash tank 16 is pumped through pipe 25 by pump 26 to the filter system.

The wax-bearing oil is pumped from storage tank 27 by pump 28 through one branch of pipe 29 into one of the gauging tanks 30, one of these tanks being filled while the other is being emptied. The oil from the gauging tanks is transferred by pump 31 to mixer 32 where it is mixed with propane, forced by pump 33 through pipe 34, and the mixture of waxy oil and propane is cooled in exchanger 35 to a temperature of about 90° F. to 100° F., this temperature being preferably selected so that the wax will be completely dissolved in the diluted oil. The mixture of wax and diluent is then selectively introduced through one of the pipes 36 into one of the chill chambers A, B, C, or D. When one of these chambers is about one-third to three-fourths full the flow thereto is cut off and the propane-oil mixture is introduced into the following chamber. While this mixture is being charged into the tank the vapor outlets 37 and 38 are preferably closed so that no appreciable cooling by evaporation of diluent takes place, and the tanks are warmed up prior to being filled so that "shock chilling" will be avoided.

The cooling of the oil to effect precipitation of wax in filterable form is not as simple a matter as it might appear. In the first place, oil and wax should be in complete solution in the propane before the chilling is initiated and since a chiller which has just been emptied is very cold, it is necessary to warm up this chiller before adding a new batch of propane-oil-wax solution. The bulk of wax precipitates as the solution is cooled from 100° F. to about 30° or 0° F. and cooling throughout this range is effected by the withdrawal of propane vapors by means of chilling compressor 21. At this time cold "used wash propane" is introduced into the chiller through line 39 to make up propane lost through vaporization and to supply additional refrigeration. The cooled slurry should have about two volumes of propane per volume of oil when the temperature reaches —40° F. After the introduction of the cold used propane the evaporative refrigeration is continued, the propane vapors being discharged through line 40 into compressor 21 and thence to the condensers 24 and back to storage chambers 12. When the temperature has been reduced to about —40° F. the vapor outlet conduit 37 is closed and outlet 38 is opened to vent the chill

chamber to the suction gas line and the holding compressor.

At about —40° F. the wax slurry is withdrawn through one of the pipes **41**, pump **42**, line **43**, and one of the feed pipes **44** to filter L, M or N. The filters which are diagrammatically shown in the drawings are vertical cylinders containing a plurality of stationary filter leaves, but it should be understood that we may employ continuous filters or filters of any other type which are known to the art. The transfer pump **42** should be of a type which will not unduly macerate or break up the wax crystals and, in fact, it may be desirable to employ fluid pressure to charge the filters or to maintain the chillers and filter supplies at a sufficiently elevated position so that the liquid head will afford the necessary filter pressure. Filtrate is withdrawn from the filters by means of one of the pipes **45**, each of which is connected with header **46**. The clear filtrate is then conveyed by gravity through pipes **47** to filtrate receiver **48**. When a sufficient cake is deposited on the filter elements the oil mixture is introduced into the next filter or bank of filters and the cake in the first filter is washed with propane from wash tank **16**, line **25**, pump **26** and one of the feed pipes **49**. When the filters are being washed with propane the so-called "wash propane" filtrate passes by gravity through pipes **50** to "used wash tank" **51** from which it is transferred by pump **52** into pipes **39**. The propane in the "used wash tank" **51** is maintained at —40° F. by the vaporization of a part thereof, the vapors being removed through pipe **53** to line **18**.

We connect the filters to line **18** by valved pipes **54**. These valves are closed during actual filter operation, but for purposes of cooling the filter or removing the filter cake, the valves may be opened.

Wax is removed from the base of the filter in the manner hereinafter described through one of pipes **55**, thence through pipe **56** to wax pumping drum **57**. The wax lines are preferably large conduits provided with screw conveyors although in some instances the wax slurry may be pumped by ordinary methods. Wax from drum **57** is forced by pump **58** into high pressure heater **59** which is maintained under a pressure of about 200 pounds gauge. Steam coil **60** maintains a temperature in the heater of about 235° F. Liquid wax, together with some propane, is withdrawn from the base of the heater through pipe **61** and passes through regulating valve **62** into atmospheric flash drum **63**. Valve **62** is automatically controlled to maintain a constant liquid level in heater **59**. The propane vaporized in wax heater **59** is withdrawn through pipe **23** which leads directly to condensers **24**. Propane from atmospheric flash drum **63** is withdrawn to line **18**.

The wax from the base of the flash chamber is withdrawn in amounts controlled by liquid level operated valve **64** and introduced into wax stripper **65** in which steam or other suitable stripping gas may be injected. The steam from the top of the stripper is condensed by the water in jet condenser **66**, the liberated propane being vented to propane line **18**. Finally the wax which has been freed from oil and propane is forced by pump **67** through cooler **68** into storage tank **69**.

Returning to the filtrate in tanks **48**, this filtrate is withdrawn through one of pipes **70** and pump **70a** to heat exchanger **15** wherein it cools the incoming propane which is to be used in washing the filters. The filtrate then passes through heat exchanger **71** and heat exchanger **72** to the top plate of the oil heater **73**. Heat may be supplied thereto by steam coil **74** so that a temperature of about 235° F. at a pressure of 200 pounds is maintained in the heater. Oil is withdrawn from the base of the heater by pipe **75** through a regulating valve **76** to low pressure flash drum **77**. Regulating valve **76** is automatically controlled to maintain a constant liquid level in heater **73**. Propane vapors are removed from the flash drum to line **18**. The oil from the base of the flash drum is passed in amounts regulated by liquid level control valve **78** into steam stripping chamber **79** where it is stripped with steam or other suitable gas. The steam is withdrawn through exchanger **71** and thence to jet condenser **80**,—the liberated propane being returned to line **18**.

The oil from the base of the stripper **79** is withdrawn through heat exchanger **72** and to oil storage tank **81**.

It will be observed from the above description that the chill chambers, unused wash propane, used wash propane, filtrate receivers and wax pumping drum are all maintained at temperatures slightly below dewaxing temperatures by the direct vaporization of propane, these vapors being withdrawn by propane line **18** and compressed in compressor **19** to about 200 pounds. It will be understood that suitable valves or pressure regulating means will be installed between the various tanks, filters, chillers, etc. and gas line **18**. The chill chambers, filtrate receivers and wax pumping drum will tend to have a temperature, substantially equal to the dewaxing temperature, while the unused wash propane and used wash propane will have a temperature slightly lower than the dewaxing temperature on account of the vapor pressure of pure propane being higher than that of a mixture. Temperatures may be controlled, however, by regulating the valves leading to line **18**.

If the pressure in the system exceeds about 200 pounds it will be released through pressure release valves on the stills or on the lines connected thereto, which valves are preferably connected to vent or fuel line **82**. Pressure release valves on the diluent storage tank are preferably set to open at about 165 pounds pressure and these valves are likewise connected to the vent or fuel line. We have diagrammatically illustrated a pressure release valve **83** in line **82**, but it should be understood that similar valves will be used wherever safety requires them. Pressure in the storage tanks **12** is usually about 150 pounds to 165 pounds gauge and use may be made of this pressure to blow the filter cake from the filter leaves (as will be hereinafter described), the propane gas being conducted through line **84** to the respective filtrate discharge pipes **45**, suitable bypasses and valves being provided so that propane can be used to discharge the filter cake without interfering with the normal filtrate discharge.

If desired, propane from line **23** may be bypassed into line **84** by line **85**. When gas is used to blow back the filter cake there will be a condensation of propane in the filter cloth due to its cold temperature, and there will be an almost instantaneous boiling or revaporization of this propane due to the low pressure on the other side of the filter cake and, as a result, the filter cake is quickly and thoroughly removed from the filter cloth and the cloth is washed by the reversed flow of propane. The same end may be accomplished

by the reverse flow of a slight amount of used wash propane liquid.

The filters may be either of the vertical cylindrical batch type diagrammatically illustrated in the drawings or they may be of the continuous pressure rotary type. In either case they will be designed to withstand considerable pressure, they will be heavily insulated and they will be provided with means for steaming or washing out the propane therefrom prior to opening for repair or replacement so that there will be no hazard from escaping propane gas.

Filtration is continued until a cake about ½" to 1" is built up on the leaf, at which time the flow of the wax slurry is stopped by closing outlet valves in line **45**, and the wash propane from tank **16** is introduced at the top of the filter through line **49***a*. The flow of this liquid propane is continued until all of the unfiltered oil in the filter is forced back (through by-pass **42***a*) to the chillers by displacement. The flow of the wash propane is then changed so as to enter the filter at the bottom and to pass through the wax cake and the filter leaves and out through line **45**. The flow of this propane is continued until practically all of the oil is washed out of the wax cake. The first wash propane may contain large amounts of oil and it may therefore be discharged with the filtrate into filtrate receivers **48** through pipes **47**. Later amounts of wash propane are removed from the filter leaves through pipe **50** to "used wash propane" tank **51**. Propane from the latter tank, as stated above, is used for diluting incoming waxy oil in chillers A, B, C, etc.

Finally the flow of wash propane from tank **16** is stopped and the remaining propane liquid is pumped from the bottom of the filter back to wash tank **16**. During this operation a small pressure is maintained inside the filter (outside of the leaves) by means of vapors admitted through lines **84** and **54**. To remove the filter cake we may employ a reverse flow of wash propane or we may employ gas from line **84**. In either case, liquid propane will be formed in the filter medium and it will be violently expanded by opening the valve in pipe **54**, this expansion causing the immediate and thorough removal of the cake from the leaf. The filter cake is directed by the hopper bottom **88** into an enlarged conduit **56** wherein a screw conveys it to wax sump **57**. If desired, a spray of liquid propane may be used to assist in washing the outside of the leaves.

Precoats or filter aids may be employed to increase filter rates and prevent clogging. Some oils are readily dewaxed with the ordinary filter elements, but other oils show a "cloud" at low temperatures after dewaxing. It is highly desirable that lubricating oil should be "zero-bright." Therefore, in some cases we make a slurry of infusorial earth or other filter-aid in propane or light oil, and apply a precoat to the filter by spraying or by passing some of the liquid through the filter. Other fibrous or crystalline filter aids may be used, either as a precoat or admixed with the wax slurry.

In the chilling step we may simultaneously remove propane vapors and add propane liquid to the chillers, we may cool by evaporating propane and subsequently adding more cold propane, or we may add all of the propane before we start to chill by evaporation. Our preferred method of cooling and of operating the filter system will now be described in connection with Figure 2.

In the modification shown in Figure 2, the propane which is withdrawn from storage is first passed through line **106** to water trapout tank **107** from which the water is withdrawn through line **108** in amounts regulated by liquid level controlled valve **109**. Propane from trap **107** is withdrawn through line **110** to wash tank **16** and through line **111** to pump **33**, line **34** and mixer **32** as hereinabove described. Any water or other immiscible liquid impurity is thus separated from the propane before it is discharged into the plant.

The next distinguishing feature of Figure 2 is the arrangement and operation of the chilling system. The warm propane-oil-wax mixture from cooler **35** and line **36** is introduced through lines **112** to one of a plurality of chillers A, B. In actual commercial use we have found that three of such chillers give excellent results, although it should be understood that any number may be used. The upper parts of these chillers are connected through gas line **113** so that if chiller A has just been emptied and chiller B has just been filled, the vapor line **113** between these chillers may be opened and propane may be boiled out of chiller B and condensed in chiller A thereby warming the latter chiller sufficiently to prevent shock chilling when the propane-oil-wax solution is introduced into it. When equilibrium has been established between the two chillers, this vapor line is closed, and propane vapors are removed from the filled chiller through lines **37** and **40**. When the temperature in this chiller, for instance chiller B, reaches about 0° to 30° F., cold used propane is introduced into it through lines **39** and **112**. The final chilling is then effected by evaporative cooling and when a temperature of −40° F. is reached the wax slurry is withdrawn by opening line **41** and withdrawing the cold slurry through main **114**, pipe **115**, main **116** and pipes **117** to one of the filter supply tanks **118**. As soon as chiller B is thus emptied the vapors from chiller A are condensed into it through line **113** until it is warmed up and then additional propane-oil-wax solution is introduced into it through line **112**.

From the above description it will be seen that the slurry is first chilled by evaporative cooling, preferably at a rate of about ½° to 4° F. per minute, it is then chilled and diluted by means of used wash propane and it is finally chilled by evaporative cooling and dropped into the filter supply tank. Before transferring the slurry from chiller B to filter supply tank **118**, we preferably connect the vapor spaces of these two tanks through line **119** so that they will be in equilibrium both as to temperature and pressure when the slurry is transferred and so that there will be no shock chilling. Lines **120** are also connected to the top of the filter supply tanks and are connected to each other through line **121** so that this system may be used to equalize the pressure on filter supply tanks as well as to return cold slurry from the filter system back to the filter supply tanks. The multiplicity of pipes and valves need not be described in detail, but it will be apparent that each chiller may be connected to chilling compressor **21** through lines **37** and **40**, they may be connected to holding compressor **19** through line **38**, they may be connected to each other through line **113**, or they may be connected to the upper part of filter supply tanks through lines **119**. The warm propane solution may be introduced into each chiller through line **36** and cold used propane may be introduced thereto through line **39**. Slurry from any chiller may be selectively introduced into any filter supply tank and slurry from the filter sys-

tem may be returned to any desired filter supply tank.

Referring now to the filter system, we have again diagrammatically shown the use of vertical batch filters L, M, LL and MM. It will be observed that the first two of these filters are connected in parallel to operate as one bank while the second two are connected in parallel to operate as another bank. We preferably have four filters in each bank, but it should be understood that any number of batch or continuous filters may be employed in either bank. Slurry from filter supply tanks 118 is transferred through line 117, line 116, line 122, pump 123 and line 124 to wax discharge line 56, which is used as a conduit for introducing this slurry into the base of the filters. When continuous filters are used, it will be understood that line 124 will lead to the wax slurry reservoir in the filter chamber. For charging wax slurry to the other bank of filters, the slurry is passed through line 125, pump 126 and line 127 to line 56A, which likewise serves as a wax discharge pipe.

When a filter cake has been built up on the filter elements and it is desired to wash the oil out of said cake, fresh cold propane from wash tank 16 is introduced through line 25, line 128, pump 123 and line 124 to filters L and M or through line 129, pump 126 and line 127 to filters LL and MM.

When it is desired to withdraw wax slurry from the base of the filters, this material may be withdrawn through line 56, line 130, pump 123, line 124 and line 131 through which it may be returned to the filter supply tanks 118. Likewise, slurry from the base of filters LL or MM may be withdrawn through line 56A, line 130A, pump 126, line 127 and line 131A which joins with line 131 and returns to the filter supply tanks. If it is desired to empty wash propane out of the filters, the wash propane is withdrawn through line 56, line 130, pump 123, line 124 and line 132 to line 25 which leads to wash tank 16. Similarly, wash propane from filters LL and MM may be passed through line 56A, line 130A, pump 126, line 127 and line 132A, which leads into line 132 for returning the wash propane to wash tank 16.

If it is desired to introduce wax slurry or wash propane into the filters at the top instead of at the bottom, this may be accomplished by opening the valve in line 133 so that pump 123 may force the liquids through line 124 and then through line 133 which acts as a manifold to which filters L and M in the first bank are connected. Similarly, pump 126 can force liquids through line 127 and line 133A into the upper parts of filters LL and MM.

Manifold 133 is connected by line 134 with low pressure gas line 18 and this low pressure gas line is likewise connected to manifold 133A through line 134A. Thus, positive connection may be made between the low pressure gas line and the shell of any filter through lines 54, 133 and 134 or lines 54A, 133A and 134A.

Manifold 133 is also connected to high pressure or blowing gas line 84 through line 135 and manifold 133A is connected thereto through line 135A.

For removing wax cake from the filter leaves it is desirable to blow back with propane gas. The filter leaves are connected to the low pressure gas line 18 through lines 136 and 137 which lead to manifold 46 and lines 136 and 137A which lead to manifold 46A. The high pressure or blowing gas line 84 is connected through lines 138 and 139 to manifold 46 and through lines 138 and 139A to manifold 46A.

It is sometimes desirable to recirculate slurry from the filters back to the filter supply tank and for this purpose we employ line 140 which is connected to manifold 133 through line 141 and to manifold 133A through line 141A. When the filter operation is just beginning and the cake is being formed on the leaves, it may be desirable to have valve 142 open, but as soon as the cake is built up and a clear filtrate is being obtained valve 142 may be closed so that the pump which is forcing the slurry into the filter may build up a pressure of about 50 pounds per square inch on the filtering surface. If the pressure exceeds this value, valve 142 may be slightly opened.

The filter leaves in filters L and M are connected through lines 45 and 46 to filtrate line 47 and filtrate collector 48; similarly, filtrate from filters LL and MM pass through lines 45A, 46A and 47. Cloudy filtrate from manifold 46 may be passed through lines 143 and 144 to line 131 through which it may be returned to the filter supply tank. Likewise, cloudy filtrate from manifold 46A may be passed through line 143A to 144 and thence back to the filter supply tank. Alternatively, the cloudy filtrate may be passed from manifold 46 through line 50 or from manifold 46A through line 50A back to the used wash tank 51.

The operation of our filter system may be described as follows: The filters are first cooled, preferably by passing cold oil through them or evaporating propane in them. Chilled wax slurry is then forced through line 116 by pump 123 through lines 124, 56 and 55 into filters L and M. The slurry is allowed to fill the filters and to overflow through lines 54 into manifold 133, thence through recirculating lines 141 and 140 back to the filter supply tanks. The initial filtrate may be cloudy and it may therefore be discharged from manifold 46 through lines 143 and 144 back to the filter supply tank. As soon as the sight glasses on the filtrate lines indicate that it is clear, the valve in line 143 is closed and the valve to line 47 is opened allowing the filtrate to be passed to the filtrate collectors 48. At this time the valves in line 54 are closed so that the pressure is gradually built up in the filters to about 50 pounds per square inch. In about 20 minutes a cake of about ½" to 1" thick is built up on the leaves.

When the cake of desired thickness has been built up, the wax slurry in the filters is displaced, preferably by propane gas from lines 84, 135 and 133, the valves in lines 54 being opened for this purpose. The displaced wax slurry is withdrawn through line 56 and may either be forced back to the filter supply tank through line 130, pump 123, line 124 and line 131 or may be forced directly back to the filter supply tank through lines 56, line 124 and line 131 by the propane pressure. The pressure of the propane gas maintains the wax cake on the filter leaves and although a small amount of the propane is condensed by the cold wax cake we have found that in a short time interval the condensation is not appreciable.

As soon as the wax slurry has been displaced from the filters, cold fresh propane from wash tank 16 is forced through line 25, line 128, pump 123, line 124 and line 56 into the filters for washing the oil out of the filter cake on the leaves. The first wash liquid may contain a large amount of oil and practically no wax so that it may be passed through manifold 46 and line 47 to the

filtrate collector. The bulk of the wash propane is, however, passed through manifold 46 and line 50 to the used wash propane tank, from which tank it is withdrawn at intervals to supply the chillers as hereinabove described. When the oil has been washed out of the wax cake, the valves in lines 54, which were closed during the washing step, are now opened again and propane gas is employed to displace the propane from the filters back to the fresh cold propane tank 16. This, again, may be accomplished by direct gas pressure or it may be accomplished by means of pump 123 and lines 124 and 132.

Instead of employing propane gas to displace the wax slurry, we may introduce liquid propane through line 133 from the top of the filters while we are displacing wax slurry from the base thereof through lines 124 and 131 back to the filter supply tank. Instead of introducing wash propane at the top and displacing the slurry from the bottom of the filters, we may merely substitute the wash propane for the wax slurry which is being introduced at the base of the filter and we may return the wax slurry to the filter supply tank through the recirculating line 140. Other modifications in operating procedure will be apparent from the detailed arrangement of valves, lines and pumps shown in Figure 2.

When all of the wash liquid has been displaced from the filter in the presence of sufficient propane gas pressure to hold the cake on the leaves, propane gas from lines 84, 136, 137, 46 and 45 is intermittently blown back from the filtrate side to cause a distending of the filter element and the removal of the wax cake therefrom. We may employ liquid propane for removing the wax from the cakes, but this is undesirable because it necessitates an undue amount of propane recovery. The wax cake which is discharged from the filter elements is conveyed by screw conveyors in line 56 to wax sump 57, as hereinabove described. This bank of filters is now ready to repeat the filtering operation. The other bank of filters operates in the same manner and further description is deemed unnecessary.

We have already described the trap for removing water from propane entering the system, but since steam is used to strip propane gas in the low pressure oil and wax stills, it is necessary to use special precautions to keep additional water from entering the system. The propane gas from the steam stripping stills is introduced through line 146 into jet condenser 66 and water is introduced through line 147 at the upper part thereof. The water and condensed steam are withdrawn from the base of this jet condenser through line 148 and we preferably employ a liquid level controller 149 which may be a simple float which mechanically or electrically operates the discharge valve in line 148. Propane gas from the upper part of the jet condenser is withdrawn by compressor 150 and forced through line 151 to vent 82, high pressure gas line 23 or low pressure gas line 18.

Another feature of our system is the use of a liquid trap 152 in line 18 on the suction side of holding compressor 19. A constant liquid level may be maintained in this trap by a suitable float control which operates valve 153 in liquid discharge pipe 154. This liquid will usually consist of propane and it may be returned to one of the diluent storage tanks or to the chiller, filter supply tanks or other cold propane tanks.

The supply of propane gas for blowing the presses is preferably obtained from the upper part of the propane storage tanks because the gas therein contains a larger amount of ethane or a larger amount of air or inert gas and therefore is less condensible. The pressure in line 84 may be regulated on the filter side of a pressure reducing valve, as illustrated in Figure 1. The filtration pressure has been described as 50 pounds per square inch, but we may employ pressures as high as 100 or 150 pounds per square inch.

The filters are provided with connections through which they may be washed out at intervals with a warm wax solvent, but since the drawings are already quite complicated, the additional wax solvent washing line will not be illustrated. Likewise, the filters are preferably provided with steam connections so that they may be steamed out prior to being opened. Other modifications in operating procedure will be apparent from the detailed arrangement of valves, lines and pumps shown in Figure 2.

It is desirable in any dewaxing process that rapid and efficient separation of oil from wax be obtained and this in turn necessitates a careful regulation of wax crystallizing and filtering operations. The wax should be completely dissolved in propane before chilling is commenced and at no time subsequent to this complete solution step should the mixture be shock chilled, i. e., come in contact with any liquid or equipment at a much lower temperature or be suddenly released from a high pressure into a zone of substantially lower pressure.

We have found that the chilling should be effected in three steps: First, the vapor space of a chiller containing warm solution should be connected to the vapor space of a cold chiller which has just been emptied so that the solution is chilled by evaporative cooling as the pressures are equalized. Cooling may then be continued by operating the variable compressor 21 until a temperature of 30° F. to 0° is reached. Next, the cold wash propane (propane which may contain some wax or oil) is added at a temperature which is lower than that of the wax mixture, but which is not low enough to produce shock chilling. This makes it possible to utilize effectively the refrigeration available through heat exchange and minimizes the volume of propane which must be distilled and recondensed. The final chilling to about —40° F. is effected by evaporative cooling by means of variable compressor 21. While we prefer to use this method of cooling, it should be understood that the cooling may be effected by conventional heat exchangers or any other means without losing other advantages of our invention hereinabove and hereinafter pointed out.

We have found that it is very desirable to filter the crystallized wax as soon as possible after it has been chilled to filtering temperature. If the chilled slurry is held for too long a time in filter supply tanks 118, we have found that filtration is more difficult and that yields are decreased. The filter supply tank should be well insulated so that none of the solidified wax will be redissolved and all valves leading to and from the supply tank should be extremely tight so that there will not be any leakage through filter valves which would cause shock chilling and which would thereby impair filter rates. We have found that a reciprocating block valve pump is quite satisfactory for transferring slurry from the filter supply tank to the filters, but even better results may be obtained by employing fluid pressure, such as compressed nitrogen, ethane or other gas, for forcing

the slurry into the filters. Where continuous filters are employed and a relatively low pressure differential is sufficient, we mount the chillers and filter supply tanks at a higher level than the filters so that the liquid head, due to difference in elevation, will be sufficient to force the diluted oil through the filter element.

Some light distillates may tend to give a flocculent wax cake which is difficult to filter, giving poor filter rates and low oil yields. To such distillates we may add a small amount of a heavier lubricating oil stock, a small amount of asphaltic material or a wax crystallization regulator of the type prepared by condensing chlorinated wax and naphthalene with aluminum chloride. Amounts of the latter material as low as .3% have been found to increase dewaxed oil yields on S. A. E. 20 Mid-Continent distillate from 70% to 84% and at the same time have been found to double filter rates.

An important feature of our filter structure is the extreme flexibility of its connections so that a single pump and a minimum of conduits may be employed to transfer all types of liquids and slurries to and from the filters of the several storage and supply tanks. Another important feature of the filter structure is the fact that each of these filters is provided with a line **155** for introducing kerosene for washing out the filters before opening them and/or for introducing steam to blow out all traces of propane. From a safety standpoint, it is highly necessary that precautions be taken to prevent the formation of explosive oil-propane mixtures.

While we have described in detail preferred embodiments of our invention, it should be understood that we do not limit ourselves to 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. In a propane dewaxing system wherein a waxy oil is dissolved in propane at elevated temperatures and pressures, cooled to solidify the wax in readily separable form, mechanically separated into wax and diluted oil and finally freed from the propane diluent, a filter vessel containing a filter element, a conduit for withdrawing wax slurry from said filter vessel, positive means for moving wax through said conduit and means for forcing a wax slurry into said vessel through the same conduit through which wax is removed therefrom.

2. In a propane dewaxing system wherein a waxy oil is dissolved in propane at elevated temperatures and pressures, cooled to solidify the wax in readily separable form, mechanically separated into wax and diluted oil and finally freed from the propane diluent, a closed pressure chilling tank, a filter supply tank connected with said chilling tank, a closed filter vessel, a filter element in said vessel, a fresh cold propane tank, a filtrate collector means including a pump and inlet connections therefor separately connected to said cold propane tank and said filter supply tank, respectively for selectively introducing into said filter a slurry from said supply tank or fresh cold propane from said fresh cold propane tank, means for returning slurry from said filter vessel to said filter supply tank and means for selectively transferring liquid from said filter element to said filtrate collector or said filter supply tank.

3. The apparatus of claim 2 which includes a used wash propane tank, means for transferring liquid from said filter element to said tank, and means for transferring liquid from said tank to said closed pressure chilling tank.

4. The apparatus of claim 2 wherein the filter element is also selectively connected to a blow back line leading from a source of propane gas.

5. In a propane dewaxing system wherein a waxy oil is dissolved in propane at elevated temperatures and pressures, cooled to solidify the wax in readily separable form, mechanically separated into wax and diluted oil and finally freed from the propane diluent, the step of periodically washing the separation means with kerosene for removing propane and improving separation rates.

6. In apparatus of the class described, a propane storage tank, a chiller, a filter, a filtrate collector, a low pressure gas line, means for mixing propane from said storage tank with waxy oil, and for introducing said mixture into said chiller, means for venting said chiller to said low pressure gas line to provide evaporative cooling in the chiller, means for introducing slurry from the chiller to said filter, means for transferring filtrate from the filter to the filtrate collector, a connection from said filtrate collector to said low pressure gas line, a propane recovery system and means for passing filtrate from said collector to said recovery system, means for stripping propane from oil with steam in said recovery system, means for condensing steam from said stripping step, separating propane therefrom and returning the propane to said low pressure gas line, a compressor and condenser connected between said low pressure gas line and said propane storage tank, a liquid trap interposed immediately in front of said compressor for trapping out and removing any condensate which may collect in that part of the propane gas line connected to the inlet side of the compressor, an inlet to said trap connected with said low pressure gas line, a connection from the upper part of said trap to the inlet of said compressor, and means for withdrawing liquid from the lower part of said trap.

7. A propane dewaxing system which comprises a propane storage tank, a chiller, a filter supply tank, a cold propane storage tank, a used propane storage tank, a filter, a filtrate receiver, means for mixing propane from said storage tank with waxy oil and for introducing said mixture into said chiller, means for transferring cold slurry to said filter supply tank, a pump with its inlet selectively connected to said chiller, filter supply tank, cold propane tank and filter shell, and with its outlet connected to said filter shell, means for passing filtrate from said filter selectively to said filtrate collector, to said filter supply tank or to said used propane tank, means for passing propane from said used propane tank to said chiller and means for passing liquids from said filter to said filter supply tank respectively.

8. The system of claim 7 which includes a vent line and a propane blow-back line both selectively connected to said filter.

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