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A. H. SCHUTTE

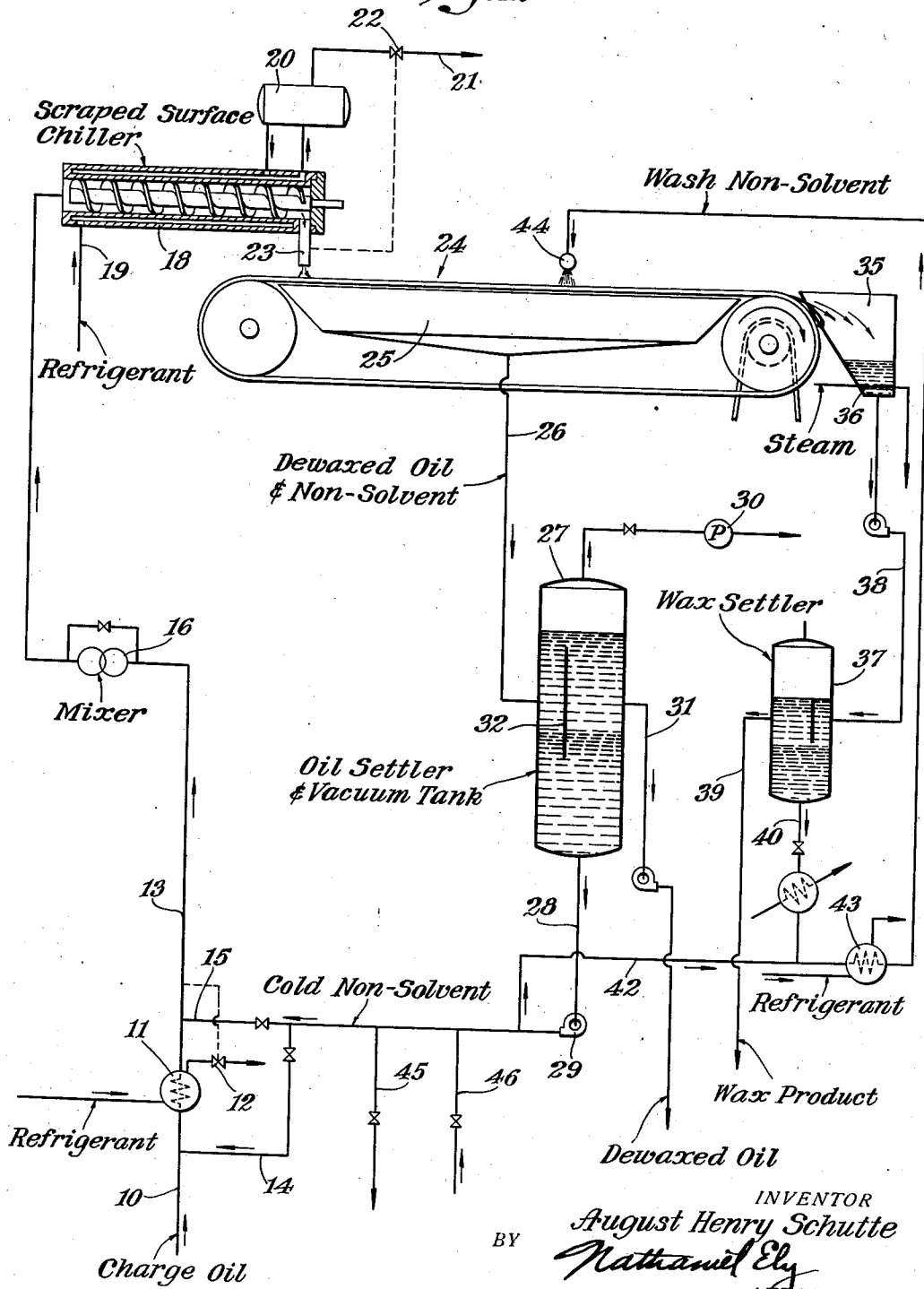
**2,168,143**

## OIL DEWAXING

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2 Sheets-Sheet 1

*Fig. 1.*



INVENTOR  
BY *August Henry Schutte*  
*Nathaniel Ely*  
ATTORNEY

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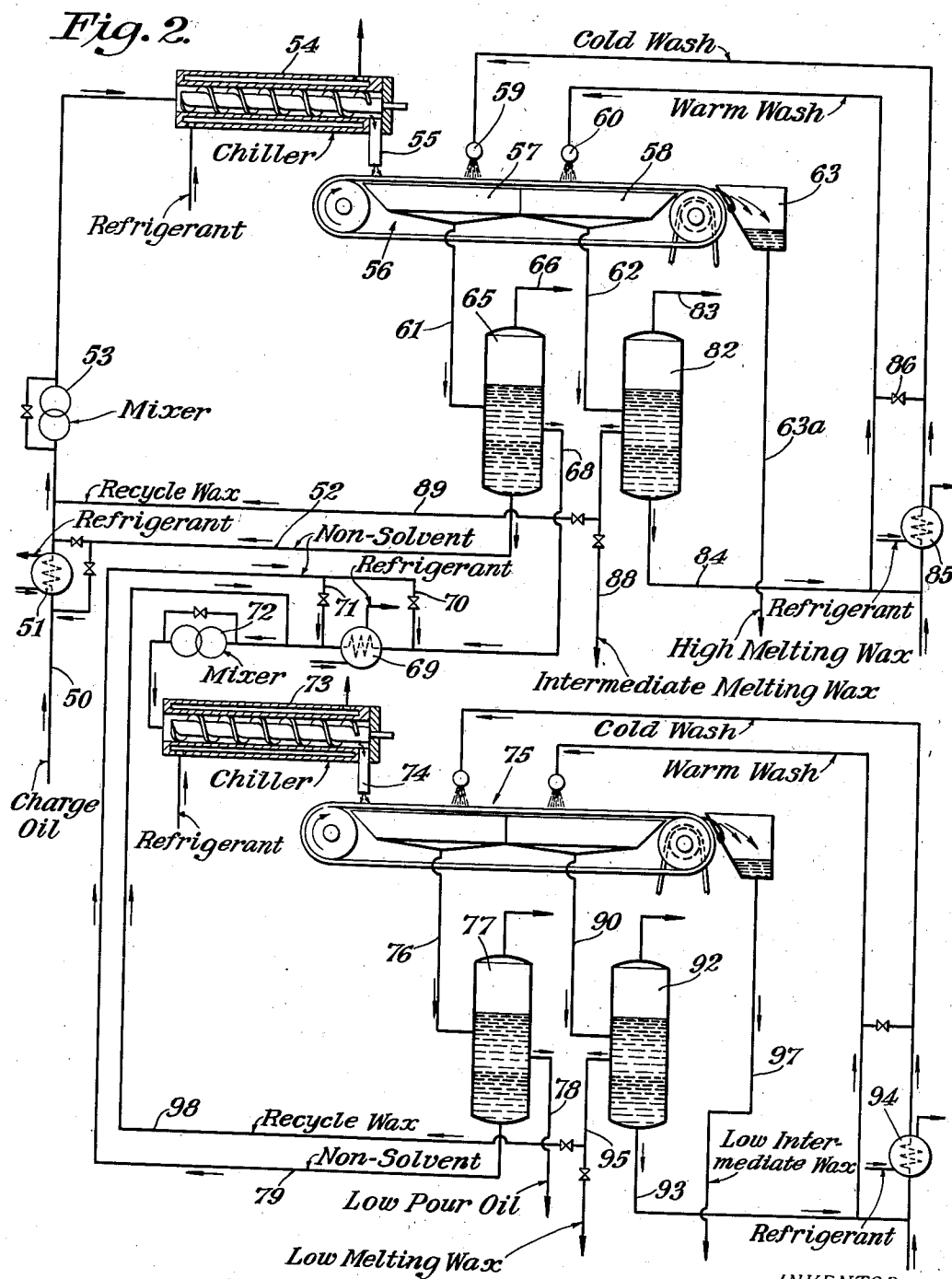
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BY

INVENTOR  
*August Henry Schutte*  
*Nathaniel Ely*  
 ATTORNEY

## UNITED STATES PATENT OFFICE

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## OIL DEWAXING

August Henry Schutte, Westfield, N. J.

Application May 19, 1938, Serial No. 208,912

8 Claims. (Cl. 196—18)

This invention relates to improvements in the art of dewaxing oils such as petroleum oils. It is an improvement on my prior invention disclosed in application Serial No. 192,534, filed February 25, 1938.

One of the principal objects of the present invention is to continuously remove wax from oils, particularly petroleum lubricating oils with a relatively low cost of utilities and added materials, and with a relatively high yield of a low pour oil having a low spread between the pour point and the dewaxing temperature.

A more specific object of the invention is to remove the wax from neutral oils containing wax of crystalline character without the use of solvents and with a minimum of refrigeration by the preliminary formation of a mixture of the neutrals with an immiscible non-freezing foreign liquid such as brine.

A further object of the invention is to continuously treat the wax as it is removed from the oil so that it is possible to simultaneously separate the wax fractions of different melting points without the necessity of a sweating operation.

Further objects and advantages of this invention will appear from the following description of preferred forms of embodiment thereof taken in connection with the attached drawings in which:

Figure 1 is a diagrammatic layout of apparatus for the removal of wax from oil; and

Figure 2 is a diagram of modified apparatus for the treatment of wax bearing oil.

I have heretofore discovered that it is possible to remove wax from petroleum oils by the intimate mixture therewith of an immiscible liquid such as water, after which the mixture is chilled and filtered. Not only is the filtration entirely satisfactory, but the economy of the process is superior to that of heretofore known methods for this purpose.

In accordance with the present invention, I find that added economies can be obtained on certain classes of oils which contain substantial amounts of crystalline wax due to the fact that a crystalline formation of the non-solvent is not essential to the free filtration of the immiscible liquid and oil. As the heat of fusion of the non-solvent need not be removed, this materially reduces the refrigeration load. Further, no heat need be supplied to separate the non-solvent from the oil by evaporation or distillation, since the separation is effectively obtained by gravity settling. More specifically, therefore, this inven-

tion relates to the treatment of neutral oils having a preponderance of crystalline wax and a relatively low viscosity. Among such oils, Diesel engine oil is a typical one but not an exclusive example. I can materially reduce the cold test of such oil by the following disclosed process. Viscous waxy oils containing commercially valuable waxes but having an oil component of poor lubricating quality may be processed cheaply for the separation of the wax by chilling only to a sufficiently low temperature to obtain the full yield of high melting point waxes.

The waxy oil charge which is introduced at 10 is first passed through a chiller 11, the temperature of which is preferably regulated by the temperature control 12, inter-connected in the chilled oil line 13. The cold non-solvent material which is of any suitable type which does not react with the oil and which does not freeze at the desired refrigerating temperature, is preferably a solution of a chemical salt such as calcium chloride brine. It is added either at 14 before chilling, or at 15 after chilling as may be desired, and depending upon its temperature.

The mixture of cold oil and non-solvent is then passed through a suitable mixing device 16 which forms an intimate mixture between the particles of waxy oil and the non-solvent. While this mixture is in the nature of an emulsion, it is not necessarily a technical emulsion. It is merely necessary that the materials be effectively comingled so that there is a fine mechanical mixture entering the chiller 18. The oil, wax and non-solvent emulsion is preferably made at about the pour point of the oil. At this temperature the oil is sufficiently viscous to produce a stable mixture.

The chiller 18 may be of any suitable type, preferably cooled by circulating a refrigerant through the walls thereof, such refrigerant entering at 19 and circulating through the receiver 20 from which the vapors are discharged at 21 for further compression. If desired, a temperature control 22 may be used inter-connected with the discharge 23 so that uniform temperature conditions are obtained and so that the chilled mixture of non-solvent and waxy oil discharged at 23 will be at the desired temperature.

The discharged chilled waxy oil and non-solvent mixture is then passed on to a suitable filter generally indicated at 24 which may be of the Fourdrinier type or of any other suitable type of centrifugal, suction or pressure, disc or leaf filter with the filtrate drawn through the filter

medium as by a vacuum applied in the suction box 25 and with the filtrate removed at 26. The filtrate is the dewaxed oil and non-solvent which is drawn into the decanting tank 27 and from which the non-solvent is removed from the bottom at 28, with the aid of pump 29, for further use. The decanting tank 27 is operated under suitable vacuum maintained by the vacuum pump 30 and the dewaxed oil is removed at 31 from the upper part of the tank.

It is found that the emulsion of wax, oil and non-solvent is readily filtered even at the low temperature to which the mass must be subjected. No diluent or solvents are necessary which accounts for the very small spread between pour point and dewaxing temperature. The non-solvent, however, is very useful in assisting filtration. In the non-solvent emulsion the waxy oil is in finely divided particles which cannot agglomerate, due to the presence of the non-oily material. The formation of a dense wax crystal structure is prevented and the filtration of the oil is thus greatly accelerated. Since each particle has been originally surrounded by non-solvent, the subsequent washing with non-solvent may be carried out at high filtration rates and with the displacement of further quantities of low pour oil. The filter cake appears as a porous easily filterable mass.

The filter cake removed from the filter 24 is discharged into the hopper 35 which may be effectively provided with a steam coil 36 so that the melted wax can be introduced into the wax settling tank 37 through the line 38. The wax product is removed from the top layer at 39 and the non-solvent, which may be carried from the filter with the wax crystals, is removed at 40 from the bottom of the settling tank 37.

During the filtration, the filter cake may be suitably washed with the non-solvent at the desired temperature, such non-solvent being provided by the line 42 from the original non-solvent supply supplemented by the non-solvent removed from the line 40, and such non-solvent is passed through a cooler 43 so that the wash temperature can be under the desired control. The wash nozzle is indicated at 44 in a suitable location on the filter. If it is found that there is an excess of non-solvent, it may be removed from the line 45, or if make-up non-solvent is desired, it can be added through the line 46. It has been found that in certain cases, the wash at 44 may be maintained at a temperature substantially higher than the chilling temperature in chiller 18 without an adverse effect on the pour of the dewaxed oil. This is of advantage since it decreases the viscosity of the filtrate during the washing step and thus increases the filtration rate. The refrigeration requirement is also thereby decreased.

The modified flow diagram as shown in Figure 2 is more particularly adapted for a higher viscosity oil containing waxes which have melting points making them commercially valuable. The oil charged at 50 is thus given a multiple step treatment to remove the wax, it being first cooled at 51, and mixed with the suitable non-solvent such as the brine heretofore described. This non-solvent in the line 52 may be added either prior to or after the cooling. The mixture is then preferably emulsified at 53 and the emulsion is then further chilled in the chiller 54 with the chilled mixture discharging at 55 onto the filter 56.

In this case, the filter 56 is provided with a plurality of suction boxes 57 and 58, each of which is adapted to receive a broad cut of filtrate and

wash liquid from wash nozzles 59 and 60. The filtrate is then removed from the respective boxes 57 and 58 through the line 61 and 62. The filter cake which is the high melting point wax, is discharged into the hopper 63 and removed to storage through line 63a.

The wash liquid in nozzle 60 is at a higher temperature than the wash at nozzle 59 so that the filtrate discharging at 62 will consist of intermediate melting point waxes and non-solvent wash liquid. Separation of the non-solvent and wash liquid is effected in the knock-out drum 65, operated under vacuum through the line 66 with the non-solvent removed at the bottom in line 52.

For the separation of the lower melting waxes, the oil is removed for further dewaxing at 68. The dewaxing steps include further chilling at 69, and mixing with additional non-solvent at 70 or 71. This mixture is then emulsified at 72 and further chilled in the chiller 73 and discharged at 74 onto the second filter 75. In this second filter, the filtrate removed at 76 from the first suction box includes low pour point oil and non-solvent. These are separated out in the knock-out drum 77 which is under vacuum. The low pour point oil is removed at 78 and the non-solvent is removed in the line 79.

In the first filter 56, the filtrate removed from the second suction box 58 is drawn into knock-out drum 82 under vacuum provided in the line 83. After settling, the bottoms product is wash liquid which is drawn off at 84 and cooled in the chiller 85 for cold wash. Some of the wash liquid may be used as warm wash and by control of the valve 86, the desired temperature can be obtained by blending the warm and cold wash for the desired temperature at the nozzles 59 and 60.

The upper layer of liquid in the knock-out drum 82 is an intermediate melting point wax removed in the line 88. This material may be returned in part in line 89 to the original charge as a recycle to increase the yield.

The filtrate removed in the line 90 from the second suction box in the filter 75 is drawn into a knock-out drum 92. The wash liquid recovered at 93 as a bottoms product may be used as the wash liquid for the second filter and, as in the first case, part passes through a chiller 94 with the portions so blended that the desired wash temperatures are obtained.

A low melting point wax is removed at 95 as another end product. If desired, a part of this may be recycled through line 98 to increase the yield.

It is thus possible to obtain not only the substantially wax free oil at 78, the high melting point wax at 63a, and a low melting point wax at 95, but it is also possible to obtain two intermediate cuts of wax at 88 and 97, the latter being the filter cake from the second filter. Recycling of the low melting point wax and intermediate melting point waxes is also provided for. The wax treating process is carried out in a continuous manner simultaneous with the oil dewaxing operation with very substantial economy and enhanced yields. It is of course to be understood that the diagrammatic showing of the drawings does not show all of the controls desirable, and the pumps necessary. Such accessories will be used under normal conditions as required by good engineering practice.

While I have shown and described preferred forms of embodiment of my invention, I am aware that modifications may be made thereto and I therefore desire a broad interpretation thereof.

within the scope and spirit of the description herein and of the claims appended hereinafter.

I claim:

1. The method of separating wax from mineral  
5 oils which comprises mixing said oil with brine to form an emulsion therewith, chilling said emulsion to precipitate the wax in finely dispersed condition, filtering out the wax as precipitate from the chilled emulsion and removing the de-  
10 waxed oil from the brine.
2. The method of removing crystalline wax from oil which comprises emulsifying the oil with a non-freezing non-solvent liquid, chilling said emulsion to precipitate the wax, filtering the  
15 chilled emulsion and removing the wax as a filter cake.
3. The method of removing crystalline wax from oil which comprises emulsifying the oil with a non-freezing non-solvent liquid, chilling said  
20 emulsion to precipitate the wax, filtering the chilled emulsion, removing the wax as a filter cake, removing the filtrate, and separating the non-solvent from the dewaxed oil in said filtrate.
4. The method of separating narrow melting  
25 point cuts of crystalline wax from oil which comprises the steps of mixing a non-solvent liquid with said oil chilling said mixture, filtering said mixture to obtain a partially dewaxed oil filtrate, mixing said dewaxed oil filtrate with additional  
30 non-solvent liquid, chilling said mixture to a lower temperature, again filtering said mixture, and obtaining a dewaxed oil as the filtrate.
5. The method of separating crystalline wax from oil which comprises the steps of intimately  
35 dispersing said oil in brine, chilling said mixture, filtering said mixture to obtain a partially dewaxed oil filtrate and a high melting point wax filter cake, mixing said dewaxed oil filtrate with additional brine, chilling said mixture to a lower

temperature, again filtering said mixture and obtaining a dewaxed oil as the filtrate.

6. The method of separating crystalline wax from oil which comprises the steps of emulsifying a non-solvent with said oil, chilling said emulsion, filtering said emulsion to obtain a partially de-  
5 waxed oil filtrate and a high melting point wax filter cake, emulsifying said dewaxed oil filtrate with additional non-solvent, chilling said emulsion to a lower temperature, again filtering said  
10 mixture and obtaining a dewaxed oil as the filtrate, and an intermediate melting point wax as the filter cake.

7. The method of reducing the cold test of light neutral oils of Diesel grade by the removal  
15 of crystalline wax therefrom which comprises the steps of adding to said oils a cold brine solution having a freezing point materially lower than the desired cold test, intimately mixing said brine and oil to form a partially stable emulsion, chill-  
20 ing said emulsion below the desired cold test of the oil to crystallize the wax, filtering the liquid from the solid constituents of the emulsified mixture and settling the brine from the removed li-  
25 quids to provide the dewaxed oil.

8. The method of reducing the cold test of  
25 light neutral oils of Diesel grade by the removal of crystalline wax therefrom which comprises the steps of adding to said oils a cold calcium chloride brine solution having a freezing point materially  
30 lower than the desired cold test, intimately mixing said brine and oil to form a partially stable emulsion, chilling said emulsion below the desired cold test of the oil to crystallize the wax, filtering the liquid from the solid constituents of  
35 the emulsified mixture and settling the brine from the removed liquids to provide the dewaxed oil.

AUGUST HENRY SCHUTTE.