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DEWAXING LUBRICANTS BY DOUBLE CENTRIFUGING

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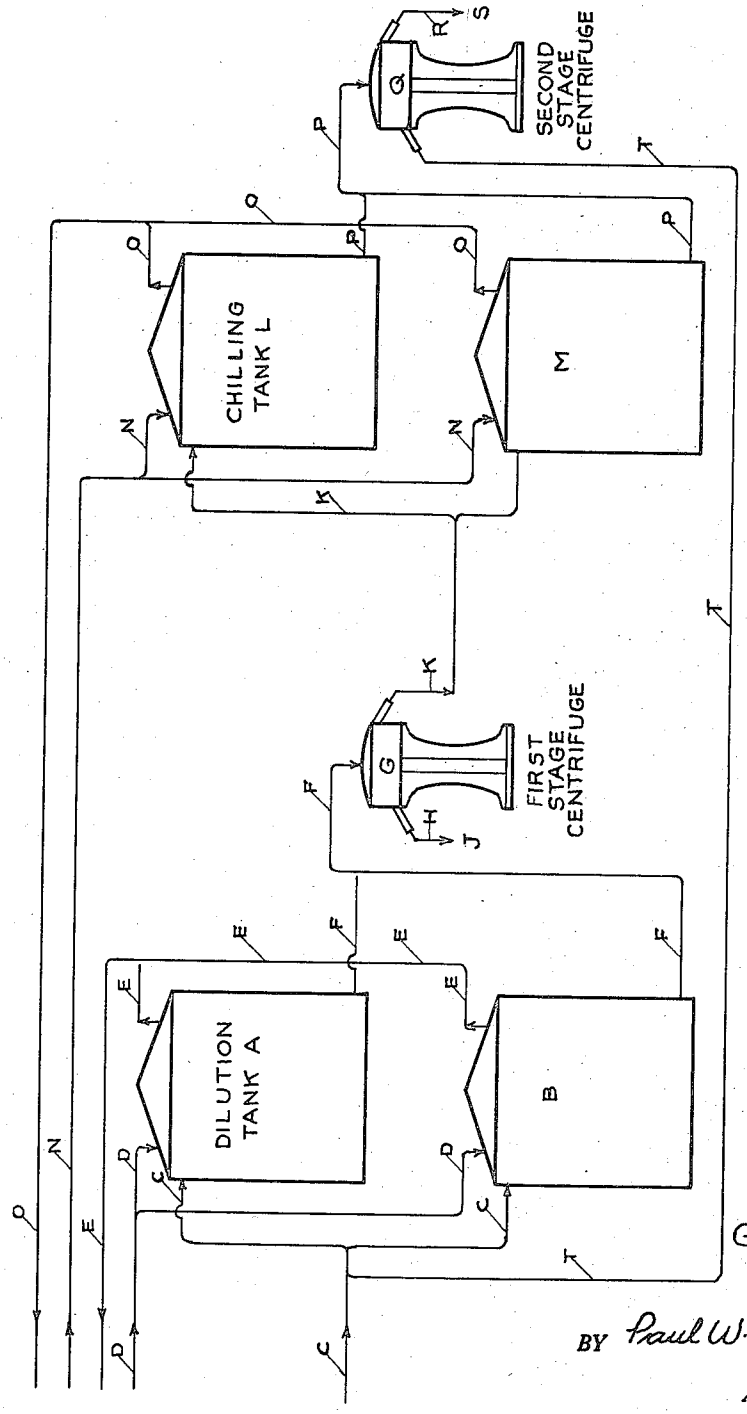


FIGURE 1

G. F. OLSEN
INVENTOR.
BY Paul W. Pritzman.
ATTORNEY.

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GEORGE FREDRIC OLSEN, OF LOS ANGELES, CALIFORNIA.

DEWAXING LUBRICANTS BY DOUBLE CENTRIFUGING.

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My invention relates to the removal of paraffin wax from lubricating oils which have been diluted with petroleum naphtha and chilled for the precipitation of the wax. My invention relates more particularly to a method in which the chilled solution is twice centrifuged at different temperatures and with different adjustments of the centrifuging machine.

The object of my invention is to obtain a drier wax than has heretofore been obtainable, thus reducing the loss of valuable oil entrained in the wax and to obtain a greater output per machine than has heretofore been possible. My invention is particularly applicable to such oils as contain a large quantity of wax and must of necessity be twice centrifuged, but it is also advantageous when applied to oils containing smaller quantities of wax and which have heretofore been completely dewaxed in one stage.

It is well known in the art that residual petroleum oils containing wax, and therefore having too high a cold test for commercial requirements, are often diluted with a suitable naphtha in order to separate the wax from the oil. The methods by which this solution of residual oils are chilled and centrifuged are also well known and understood. In general terms these methods comprise: chilling the oil to the requisite low temperature, (which is often as low as minus 20° F.) and passing it through a suitable type of centrifuge by which a finely crystalline or amorphous wax containing considerable entrained oil is separated from the solution of residual oil. The latter is then deprived of the solvent by well known methods, and sometimes further treated with acid or adsorbent, or both, for the production of the desired color.

In cases where oils contain a very large proportion of wax it has heretofore been the practice to partially chill the oil for the separation of a portion of the wax, to return the partially dewaxed solution to a second chiller by which it is brought to the final temperature, and to centrifuge the second chilled solution for obtaining a second crop of wax and the required cold test.

In my improved method of dewaxing in two stages I follow any usual or preferred method of carrying out the chilling and centrifuging operations, my invention lying, first, in the rate at which the oil is chilled in the two stages, second, in the different time rates at which the oil is centrifuged in the two stages, and third in returning the second stage wax to a new batch of unseparated oil for the recovery from said second stage wax of a substantial quantity of oil entrained therein.

In detail, my method consists of the following steps: I first prepare the customary solution of oil in naphtha or petroleum distillate, using the customary proportion of naphtha to oil, which may range from three parts of oil with two parts of naphtha to two parts of oil with three parts of naphtha. I then chill this solution, using any usual or preferred means, to a temperature ranging form 26° to 20° above zero F., at which temperature a material proportion of the wax will be set free from solution. This first stage chilling should be carried out at a rate of about 4° F. temperature drop per hour, in order to avoid gelatinization of the oil-wax solution. The chilled mixture is then centrifuged in the usual manner, but in this first centrifuging it is essential that the machine be set to a close adjustment and the driest possible wax obtained. This wax is segregated and directed to its ordinary uses, which are no part of my present invention.

The solution, still containing a small quantity of wax, is then further chilled to such temperature as is required to produce the required cold test on the finished oil. This temperature can not be stated in figures as it will depend on the nature of the oil, the degree of dilution and the predetermined cold test, but it may be said that the temperature will be substantially higher than would be required if the wax were to be removed in the first stage, or even in a two stage operation as heretofore conducted. The twice chilled oil is then again centrifuged but, instead of using the usual adjustment of the machine, this is set to a very slack adjustment which will remove the wax rapidly and completely from the solution but will leave a material quantity of solution entrained in the wax. The advantages of this slack adjustment are a much greater throughout capacity for the second stage machine, the more com-

plete removal of wax, and a higher centrifuging temperature with a corresponding reduction in the amount of refrigeration required—which is most expensive at such low temperatures as are required for the final chill.

If this second stage wax were to be segregated and diverted to its usual uses the loss of oil by entrainment in the wax would be a prohibitive bar to the application of this method. I therefore return this slack wax, which consists of paraffins having relatively low melting points, to another batch of fresh solution prior to the first chilling. On the addition of such wax to an unchilled solution the wax immediately dissolves and is in part reprecipitated in the first stage chilling of the second batch, the harder and higher melting point waxes carried by the original solution acting as points d'appui for this smaller quantity of soft wax and preventing it from assuming the slimy consistency which makes it difficult and tedious to separate sharply from the solution.

Inasmuch as all the wax is finally removed, in the first stage centrifuging, it may appear at first sight that the addition of a second stage centrifuging is a useless loading of the manipulation. This, however, is not the case. In ordinary two stage chilling it is the custom to strip the oil from the wax as completely as possible in both stages, and in single stage chilling it is absolutely necessary to strip closely in the single centrifuging operation. When this close stripping in a single stage is conducted at the very low temperature required to produce the final cold test, the separation becomes very slow because of the material increase in viscosity of the solution at such low temperatures, which range from 20 to 30 or even 40° F. below zero. If the chilling is conducted in two stages and the wax product from the second stage is segregated as wax, as has heretofore been the custom, it is necessary to strip very closely in the second stage, and this is still more tedious because of the soft consistency of the wax separated in the second stage and the relatively slight gravity difference between this wax and the solution. The density of the solution increases rapidly with diminishing temperature while the density of the wax remains substantially unchanged. If, however, the second stage wax be produced by a slack adjustment of the machine, so that it will entrain and carry away a material proportion of solution into the slack wax, which is returned to an original solution for the stripping of the oil from the wax, the second stage operation also may be performed rapidly and cheaply. The total output of a given number of machines, when handled according to my improved method as above set forth, will always be materially greater than the output which could be obtained from them by the application of the usual method of two stage chilling and centrifuging, and will often be materially greater than their output when operated single stage on oils which can be dewaxed in a single operation. The final wax will also carry less oil, the operating loss will be correspondingly reduced, and the refrigeration effect required will also be materially decreased.

As an illustration of the manner in which my invention may be applied to the production of low cold test lubricating oils and without in any way limiting the scope of such invention to the specific details comprised in such description, I submit the following:

I start with a crude petroleum of suitable quality, such for instance as the oil from the pioneer field in Oklahoma. This oil I reduce in a still to the required viscosity and flash, treat the residue or still bottom with sulfuric acid, settle out the acid sludge thus formed, and neutralize and decolorize the acid treated oil by heating it with a suitable adsorbent clay, such for instance as the well known Death Valley clay, at a temperature approximately 450° and 500° F., finally filtering out the spent clay and permittitng the decolorized oil to cool. This is ordinary refinery practice which is well known and understood and is no part of my invention, the description being given solely to illustrate the manner in which a raw stock suitable for the application of my invention may be prepared.

I then introduce any desired quantity of the prepared lubricating stock, which contains a considerable proportion of wax or paraffin, into one or the other of the dilution tanks A—B, (see Fig. 1). These tanks are provided for the purpose of diluting the oil and chilling the solution formed by such dilution, and may be identical in size and construction, the tanks being most conveniently used alternately. In the present example I introduce the prepared lubricatitng stock into tank A, through the pipe line C communicating with a source of supply of lubricating stock not shown. Pipe C also communicates with a source of supply not shown of a suitable diluting material. I prefer to use a petroleum naphtha having the general characteristics of gasoline, but a somewhat narrower cut, a fraction having an initial point of 200° F. and an end point of 400° F. being suitable. This naphtha should be refined to a water white color and should be clean and sweet. I would use about one and one-half volumes of such naphtha to one volume of the prepared lubricating stock and mix the two in tank A by any convenient agitating means not shown.

The dilution tanks A and B should be provided with cooling coils through which cold brine may be passed for the purpose of reducing the temperature of the contents of the tank. Such brine is admitted through the

pipe D from any convenient refrigerating means, passing thence through coils within the tank where it absorbs heat from the contents of the tank, and passing out through the pipe E to return to the refrigerating means. This brine may be cooled to about 0° F., and while brine is being passed through the coils the contents of the tank should be gently stirred to bring the cooling oil into contact with the coils, but the stirring should not be sufficiently vigorous to break the crystals of wax as this will cause the formation of very fine crystals which are difficult to separate. In this first stage chilling I prefer to cool at a rate of approximately 4° F. temperature drop per hour and in this specific example I prefer to cool the solution to approximately 26° F.

When this temperature has been reached the contents of the dilution tank are withdrawn through the pipe F and fed into the first stage centrifugal G. This centrifuge may be of any preferred type and there are available numerous machines adapted to this particular purpose of separating oil from wax, the character and use of which is well known to those skilled in the art.

In this machine the solution is separated from the larger part but not all of the wax. The machine should be set to a very close adjustment in order to obtain the driest possible wax as the wax is less valuable than the oil and the wax discharge from this machine is a final discharge and any oil content is irrecoverable. The wax leaves the machine through the pipe or conduit H and is discharged as at the point J to a container from which it may be withdrawn for any use for which it may be suited.

The oil solution from centrifugal G is carried through the pipe K to one or the other of a pair of cooling tanks L—M, which may be similar to or identical with solution tanks A and B. In the present illustration I will assume that the oil is discharged through the pipe K into tank L, tank M being already filled with solution resulting from a prior operation of the same character. These tanks also are equipped with cooling coils supplied with cold brine through the pipe N from a refrigerating means not shown, the warmed brine returning through the pipe O to the refrigerating means. It is obvious that by making suitable connections between pipe O and pipe D the cooling brine escaping from tanks L and M, which are maintained at a very low temperature, might be used for chilling the contents of tanks A and B, which are maintained at a materially higher temperature.

The contents of tanks L and M should be cooled to a temperature suitable for obtaining the desired flash test on the finished oil, in this case say 20° below 0° F. It will be understood that these tanks are to be used alternately, the contents of one of them being fed through pipe P to the second stage centrifugal Q, while the other tank is being filled and its contents reduced to temperature. In the present illustration, tank M is being used to feed the centrifugal while the opposite tank L is being filled and cooled. The cooling of these tanks may proceed at any desired rate which is economical of chilling effect and it will be found that it is economical and desirable to cool at a rate materially faster than the 4° per hour to which the first stage chilling is limited, as for instance at a rate of 10° to 15° F. per hour.

The second stage centrifugal Q is set with a slack adjustment and is fed at such rate that, while the wax is not completely deprived of solution, the solution is completely deprived of wax. The dewaxed solution is conveyed from the centrifugal through the pipe R and escapes as at S to a tank or receptacle from which it may be withdrawn for the removal of the diluting naphtha, by which removal the lubricating oil is recovered as such. This removal of naphtha by distilling and steaming is conventional practice and is no part of my present invention.

The slack wax from the second stage centrifugal Q still contains a valuable quantity of solution. It is, therefore, conveyed from this centrifugal through the pipes T and C into the first stage dilution tank B, where it is admixed with a new charge of oil, either before or after the dilution of such oil with naphtha, but prior to the chilling operation above described. The mixture, being diluted, is then chilled as first described, to the aforesaid temperature of approximately 26° F. The operation should be so conducted that by the time tank A is empty tank B will have been filled and cooled to the proper temperature. At such time tank L will be filled with partly stripped oil and tank M should also be empty at this time, its contents having been fed through the second stage centrifugal. At such time the valve connections to the tanks are reversed, the first stage centrifugal G being fed from tank B, the discharge from this centrifugal passing into tank M, the second stage centrifugal Q being fed from tank L and the discharge from this centrifugal passing back to tank A. By this means continuous operation of the centrifuges may be maintained, the only requisite being that the feed to the two centrifugals be balanced at substantially the same rates in order that the pairs of tanks (A—L and B—M) may empty at substantially the same time. Where I speak of a centrifugal G or a centrifugal Q it will be understood that in practice a plurality of centrifugal units would be provided for each stage, the capacity of each individual unit being small.

I call particular attention to the fact that, in this method of operating, the final removal

of wax is solely from the first stage centrifugal; that the wax from the second stage centrifugal passes back to and through the first stage, and that the final separation of dewaxed oil is from the second stage centrifugal only.

I claim as my invention:

1. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; slowly chilling the solution to substantially 20° F.; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; again chilling said solution at a substantially faster rate than in the first chilling, to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

2. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; slowly chilling the solution to a temperature of partial separation of the wax from said solution; diverting substantially oil-free wax from the system; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; again chilling said solution at a substantially faster rate than in the first chilling, to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

3. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; slowly chilling the solution to substantially 20° F.; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; further chilling said solution to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

4. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; slowly chilling the solution to a temperature of partial separation of wax from said solution; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; further chilling said solution to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

5. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; chilling the solution at a rate not exceeding 4° F. temperature drop per hour to substantially 20° F.; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; again chilling said solution at a substantially faster rate than in the first chilling, to such a point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

6. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; chilling the solution at a rate not exceeding 4° F. temperature drop per hour to a temperature of partial separation of the wax from said solution; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax, diverting substantially oil-free wax from the system; again chilling said solution at a substantially faster rate than in the first chilling, to such point as will bring

the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

7. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; chilling the solution at a rate not exceeding 4° F. temperature drop per hour to substantially 20° F.; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; further chilling said solution to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

8. The method of separating wax from lubricating oils by means of dilution, chilling and centrifuging, comprising the following steps in the order named: diluting the oil with naphtha; chilling the solution at a rate not exceeding 4° F. temperature drop per hour to a temperature of partial separation of wax from said solution; centrifuging said solution with a close adjustment of the machine for sharp separation of solution from wax; diverting substantially oil-free wax from the system; further chilling said solution to such point as will bring the finished oil to the desired cold test; centrifuging the second chilled solution with a slack adjustment of the machine for the partial separation of solution from wax; diverting substantially wax-free oil from the system; returning the last said slack wax to another batch of diluted oil which has not been chilled, and chilling said mixture of oil and wax and centrifuging the chilled mixture as first aforesaid.

In witness that I claim the foregoing I have hereunto subscribed my name this 6th day of October, 1927.

GEORGE FREDRIC OLSEN.