

## UNITED STATES PATENT OFFICE

BENJAMIN L. SOUTHER, OF PITTSBURGH, AND WILLIAM A. GRUSE, OF WILKINSBURG,  
PENNSYLVANIA, ASSIGNORS TO GULF REFINING COMPANY, OF PITTSBURGH, PENN-  
SYLVANIA, A CORPORATION OF TEXAS

## TREATMENT OF PETROLATUM STOCKS

No Drawing.

Application filed June 5, 1928. Serial No. 283,122.

This invention relates to the treatment of petrolatum stocks; and it comprises a process of separating petrolatum stock into its several component parts such as waxy matter, asphaltic material, resins and viscous oil of low cold test and low carbon content, wherein petrolatum stock, separated from cylinder stock in the ordinary way, by cold settling of heavy steam refined petroleum oils with the aid of naphtha, is dissolved in butyl alcohol to form a solution, said solution is cooled to separate products other than contained heavy oil, the separated material is removed and the mineral oil recovered from the remaining solution; cooling and settling advantageously being accomplished in a succession of steps with the separation and removal of a single component at each step; all as more fully hereinafter set forth and as claimed.

It has been common practice since early days of the petroleum refining art to remove the wax content of certain heavy steam refined petroleum oils by dissolving or diluting said oil in petroleum naphtha and chilling the mixture, whereupon the wax content separates out of solution. The waxy material so separated has long been known as "petrolatum stock" and the "petrolatum" or "petroleum jelly" of commerce has been produced by refining this material.

The described cold settling process is used in the production of heavy oils of low wax content and oils which have a low carbon residue test. Of recent years the demand for these extremely viscous, low cold test and low carbon test oils has been such as to result in the incidental production of a quantity of petrolatum stock greatly out of proportion to the market demand for petrolatum. As a result it is common to convert much of this petrolatum stock into gasoline by cracking processes.

We have found that petrolatum of the character described contains a microcrystalline wax of the general character of ceresin and, like ceresin, is useful for many technical purposes. It is accompanied by certain resinous and asphaltic or gummy bodies which must be separated in order to develop its character.

In the present invention it is the object to recover the described valuable ceresin-like wax and also high grade heavy oil as well as a resinous and asphaltic material as a by-product.

In a practical embodiment of our invention we start with the described petrolatum stock produced by cold settling heavy steam refined petroleum oils with the aid of petroleum naphtha. This petrolatum stock often carries as high as 25 per cent of the naphtha used in the cold settling process. This is first removed by steaming. To the stock so produced is added butyl alcohol. From 4 to 6 parts alcohol are added, the proportion depending somewhat on the character of the stock. Admixture of alcohol is effected at a temperature sufficient to produce complete solution of the stock in the alcohol. This temperature is advantageously around 180° F. The solution is then cooled somewhat, say to a temperature of 140° F. At this temperature the resins and asphaltic material separate out as a distinct layer which is removed by settling or with the aid of a centrifuge. The remaining solution is next chilled to about ordinary room temperature, say, to about 70° F. At this temperature a large part of the wax content separates out and this is removed by settling or with the aid of a centrifuge. The solution, now deprived of its resins and asphaltic material and of part of its wax content, is chilled, advantageously to a temperature of from 15° F. to 0° F., whereupon additional wax separates out. This is removed by centrifuging. The two wax fractions so obtained have somewhat different characteristics. The solution remaining after the last centrifuging is distilled to remove and recover the butyl alcohol. The residue is a highly viscous mineral oil

of low cold test and low carbon residue test, which is unusually free of the resins commonly accompanying such oils. The recovered butyl alcohol goes back for reuse.

5 In the event that naphtha was not separated from the petrolatum stock at first, it goes through the process and must be separated from the butyl alcohol by fractionation. It is therefore advantageous to separate the  
10 naphtha preliminarily.

The foregoing process as described takes the resins and asphaltic materials out in one cooling and separation with subsequent removal of wax in the two stages. It is how-  
15 ever possible to separate and remove all of the wax in a single stage, obtaining the two fractions together. Whether the single stage or two-stage separation is used depends upon the particular character of the wax desired.

20 The cooling may be effected in a single stage without any intermediate removal of solid matter. In this event, the resins, the asphaltic material and the wax come out together as a composite material. This may be re-  
25 fined by various processes to give material of the general character of Montan wax. The waxes separated by single stage or two-stage separation vary somewhat in melting points and other characteristics. They are  
30 high melting but have no sharp melting point. While of crystalline character when pure, the crystallinity is not as highly developed as in paraffin wax and the general properties are quite different. As stated, the  
35 wax resembles ceresin. Generally the wax is rather brown in color. For many purposes the color is not disadvantageous. Where a lighter product is wanted, the wax may be refined by acid treating and the use of clay.

40 Generally the asphaltic and resinous fractions carry considerable proportions of wax and it is sometimes worth while to repeat the process of dissolving in butyl alcohol and chilling. For example, the asphaltic and  
45 resinous material separated may be extracted several times with butyl alcohol, using each time, say, 0.8 parts by weight of the alcohol.

In a modification of this invention, after resins and asphaltic material have been re-  
50 moved and prior to the last cooling and separation of wax, acetone is added. The addition of acetone at this point effects a more complete separation of the wax and the wax is of somewhat purer character. The oil re-  
55 covered from the solution has a lower pour test.

In one embodiment of this invention, we treated a petrolatum stock produced by the ordinary cold settling with naphtha of an  
60 oil of the following tests: gravity, 23° A. P. I., at 60° F.; viscosity, 150 sec., Saybolt, at 210° F.; flash point, 510° F.; pour point, 75° F.; carbon residue test, 4.0 per cent. This petrolatum stock was steamed and sub-  
65 jected to the process here disclosed. There

was recovered about 20 per cent of heavy mineral oil having a density of 0.92, a pour test of 25° F. and a viscosity of 127 seconds, Saybolt, at 210° F. Its Conradson carbon  
70 residue test was about that of an ordinary bright stock, viz. 2.7 per cent. On the same stock and proceeding in the same manner, except for the addition of acetone before the last wax separation, an oil was obtained hav-  
75 ing characteristics the same as the oil just mentioned but the pour test was 10° lower. This reduction of pour test was effected by using 15 parts of acetone to 25 parts of solu-  
tion.

By cooling the original solution of petro-  
80 latum stock in butyl alcohol to a temperature at which only the resins and asphaltic material are thrown out a material is obtained which on distilling off butyl alcohol  
85 can be used to produce a superior quality of petrolatum freer of resins and asphaltic material than that produced by ordinary means.

While the removal of separated matter has in each case been described as being accom-  
90 plished with the aid of a centrifuge, removal can also be effected by ordinary gravity settling. The advantage offered by the use of centrifuges is that the settling time is short-  
95 ened and the separation is so improved as to permit operating the process as a practically continuous one.

The removal of wax in a single stage or in two or more stages is, as stated, to some extent an economic matter depending on the  
100 cost of centrifuging on one hand as against the ease of removal and simplification of the chilling process on the other hand. The removal of the waxes and resins separately or jointly is an economic matter, depending on  
105 their value.

Secondary butyl alcohol can be substituted in this process for the normal buytl alcohol and will give about the same results.

Where the main object is the recovery of high grade oil from petrolatum stock by  
110 the addition of butyl alcohol as described the mixture is chilled in a single stage and the separated matter removed. In this event the temperature to which the solution is cooled is dictated by the cold test desired in  
115 the recovered oil.

What we claim is:

1. The process of recovering heavy mineral oil from petrolatum stock which comprises adding a butyl alcohol to petrolatum  
120 stock at a temperature sufficient to effect solution, cooling the solution to produce a separation of asphaltic, resinous and waxy material, removing the so separated material, distilling off butyl alcohol and recover-  
125 ing the heavy mineral oil residue.

2. The process of recovering heavy mineral oil of low wax content and low carbon residue test from petrolatum stock which comprises  
adding a butyl alcohol to petrolatum stock 130

under conditions forming a solution, chilling said solution to a low temperature and effecting a separation of asphaltic, resinous and waxy material, removing the so separated material from the liquid, distilling off butyl alcohol and recovering the heavy mineral oil residue.

3. The process of treating petrolatum stock which comprises adding a butyl alcohol to petrolatum stock at such a temperature as will effect solution, cooling said solution to a temperature sufficient to separate contained resins and asphalts and enough of the wax to produce a product liquid at room temperature, removing the so separated material and distilling the butyl alcohol out of the remaining liquid to produce a liquid residue containing heavy mineral oil.

4. The process of separating petrolatum stock into its component parts and recovering wax and by-products which comprises adding a butyl alcohol to petrolatum stock at a temperature sufficient to effect solution, cooling said solution to a temperature sufficient to separate contained resins and asphaltic material while not so low as to separate wax, removing the separated material, further cooling the liquid to a temperature sufficient to separate wax, removing the separated wax and distilling the butyl alcohol out of the remaining liquid to recover heavy mineral oil.

5. The process of separating petrolatum stock into resins and asphalts, wax and oil of low wax and carbon content which comprises adding a butyl alcohol to petrolatum stock at such temperature as will effect solution, cooling said solution to a temperature sufficient to separate contained resins and asphaltic material while not so low as to separate a substantial amount of wax, removing the separated material, adding acetone to the liquid, further cooling the liquid to a temperature whereby wax is separated, removing the separated wax and distilling the butyl alcohol and acetone out of the remaining liquid to produce a residual heavy mineral oil of low wax and carbon content.

6. The process of separating petrolatum stock into resins and asphaltic material, wax and oil of low wax content and low carbon residue test which comprises adding a butyl alcohol to petrolatum stock at such temperature as will effect solution, cooling said solution to a temperature sufficient to separate contained resins and asphaltic materials while not so low as to separate a substantial amount of wax, removing the separated material, similarly removing the wax from residual liquid by cooling in a plurality of stages with separate removal of wax in each stage, adding acetone to the liquid immediately before the last stage of wax separation and distilling the butyl alcohol and acetone out of the liquid from which the

resins and asphaltic material and waxes have been removed, to produce a residual heavy mineral oil of low wax content and low carbon residue test.

7. The process of separating petrolatum stock into resins and asphaltic material, wax and oil of low wax content and low carbon residue test which comprises dissolving petrolatum stock in a butyl alcohol at a temperature of approximately 180° F., cooling said solution to a temperature of approximately 140° F. to produce a separation, removing separated material, further cooling the liquid to a temperature below 32° F., removing the separated material and distilling the butyl alcohol out of the remaining liquid to produce a residual heavy mineral oil of low cold test and of low carbon residue test.

8. The process of separating petrolatum stock into resins and asphaltic material, wax and oil of low wax content and low carbon residue test which comprises dissolving petrolatum stock in a butyl alcohol at a temperature of approximately 180° F., cooling said solution to a temperature of approximately 140° F. to produce a separation, removing separated material, adding acetone to the liquid, further chilling the liquid to a temperature below 32° F. and removing the separated material, and distilling the butyl alcohol and acetone out of the remaining liquid to produce a residual heavy mineral oil of low cold test and of low carbon residue test.

In testimony whereof, we have hereunto affixed our signatures at Pittsburgh, Pennsylvania, this 2nd day of June, 1928.

BENJAMIN L. SOUTHER.  
WILLIAM A. GRUSE.