

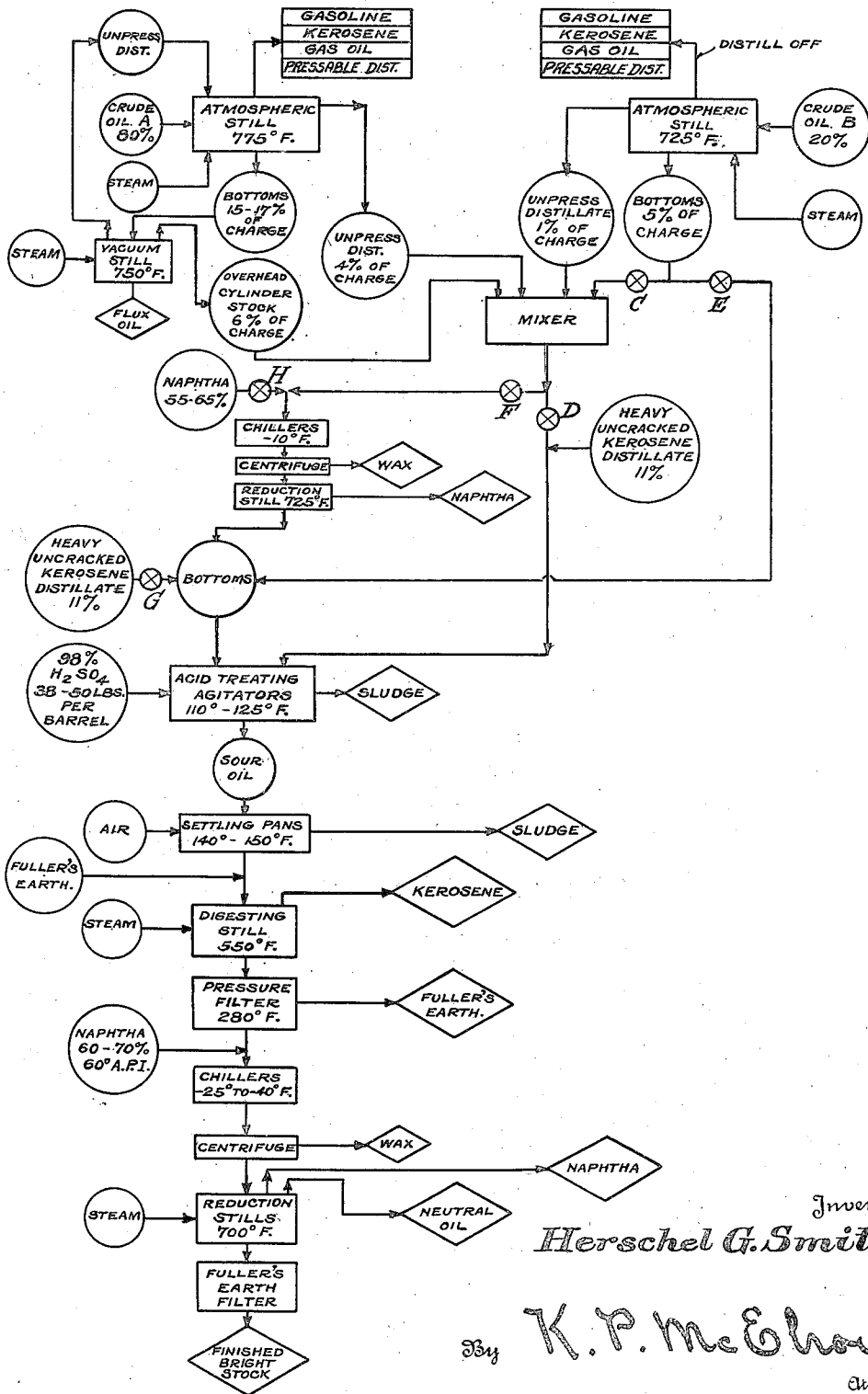
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H. G. SMITH

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MANUFACTURE OF HEAVY LUBRICATING OILS

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Inventor
Herschel G. Smith,

By *K. P. McElroy*
Attorney

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MANUFACTURE OF HEAVY LUBRICATING OILS

Herschel G. Smith, Swarthmore, Pa., assignor to
Gulf Refining Company, Pittsburgh, Pa., a corporation of Texas

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This invention relates to the manufacture of heavy lubricating oils; and it comprises a method of making heavy, blended, dewaxed lubricants from a mixture of overhead and residual lubricating stocks, advantageously derived from different crude oils, wherein such an overhead lubricating stock is mixed with such a residual stock in a ratio depending upon the viscosity range of the residual stock and on the viscosity of the overhead stock, the percentage of residual oil used being the greater the lower the viscosity of the overhead stock and the mixture is diluted with naphtha and separated in a centrifugal; and it comprises, as a new product a dewaxing stock containing a mixture of naphtha, overheads and residuals in proportions determined by the viscosities of said overheads and residuals; and it also comprises a blended lubricant stock produced by the said process, comprising a mixture of one or more heavy distillates and residuals, the proportion of residuals in said blended lubricant bearing a definite relation to the viscosity of the overheads contained therein; all as more fully hereinafter set forth and as claimed.

At the present time there is a considerable demand for viscous lubricating oils of low pour test. Such oils, after wax removal and refining, usually have viscosities ranging from 60 to 210 seconds at 210° F. by the Saybolt universal viscosimeter (S. U. V.), and pour tests from +30° F. down to -10° F., or even lower. The requirement of a low pour test in such oils has necessitated the development of special methods for the wax removal from the lubricating oil stock. The wax in such stocks may be amenable to removal by the usual centrifuging methods but frequently it occurs in a form in which it can be removed only with the greatest difficulty. Neither filter pressing nor centrifuging are feasible in the case of some of these stocks; this being especially true of many of the so-called unpressable distillates and the overhead cylinder stocks distilled from paraffin type crudes containing asphaltic materials. Alone, these distillates cannot be handled in a centrifugal.

As is well known the residuals from many paraffin and mixed type crudes can not be economically refined to lubricating oils because of the presence of excessive asphaltic material in such residuals. It is advantageous to subject such crudes to a fire and steam distillation to distill off the lubricating fractions of value, leaving as a residuum a heavy asphaltic product known as "heavy flux oil", usually disposed of as a fuel oil. The viscous lubricants are thus recovered as un-

pressable distillates and overhead cylinder stocks while most of the dark colored asphaltic material is eliminated in the flux oil. The removal of wax from these viscous distillates represents a difficult problem.

In order to circumvent this problem the art has employed various expedients, all of which are more or less wasteful. For example, it has been considered necessary in some cases to redistill the viscous distillates with a certain amount of cracking to alter the wax structure to a more crystalline type in order that it might be eventually separated by the usual pressing methods. This procedure destroys a large part of the viscosity of the more viscous lubricants by cracking them to less viscous oils.

Special methods have also been suggested for the removal of wax from unpressable distillates and overhead cylinder stocks, for example, the use of paper pulp or fuller's earth as filter-aids. All of these methods entail additional expense and none has satisfactorily solved the problem as a whole. In particular, the use of a filter-aid requires additional equipment, and somewhat expensive process steps. The removal of the added material itself from the wax cake becomes difficult and expensive.

Removal of wax by the modern centrifuge has several important advantages. But in order that such removal may be successful the wax must possess certain rather specific properties. Waxes of the so-called crystalline type, such as those encountered in light, wax-bearing distillates, can usually be removed by chilling and filter pressing but are not readily removed in a centrifugal. On the other hand the so-called amorphous types of wax which occur in viscous oils such as uncracked residuals cannot be removed economically by filter pressing. This particular type ordinarily is not difficult to remove by centrifuging. Intermediate or semi-crystalline waxes cause difficulties in both the filter pressing and in the centrifuging methods. These waxes occur most frequently in the more viscous distilled lubricating fractions, designated as unpressable distillates and overhead cylinder stocks. When an oil containing this type of wax is subjected to the usual refining steps, such as acid treatment and/or clay treatment, and is then diluted and chilled preparatory to centrifuging, the wax tends to develop a gel-like structure. Centrifugal force is not effective upon such a structure and separation cannot be secured.

On the other hand, while some separation of semi-crystalline wax can be secured by changing the standard procedure to the extent of dewaxing before instead of after acid treatment, the semi-crystalline type of wax, in the presence of the unrefined oil, tends to form hard masses of wax in the centrifuge which soon plug up the rotor of the machine. Continuous discharge of wax from the centrifuge cannot be effected by standard procedure. One of the achieved objects of this invention is to so effect the satisfactory control of the wax structure as to enable precipitation of the wax in flocculent particles that will not pack to hard masses and, at the same time, will not form gels in a chilled diluted mixture.

I have found that the readiness with which wax can be removed from a stock in centrifuging depends on the viscosity of the oils composing the stock. This is the case even when, as is usual, the viscosity of the stock is adjusted before centrifuging, by diluting with light naphtha. The viscosity of a distilled lubricating oil fraction from a given source is related to the average molecular size of its constituents and to the average boiling point of the fraction, as well as other physical properties. The physical property of viscosity in this case is merely selected for a control test, as the most convenient index of the desired properties. The specific effect of the viscosity of an oil upon the physical properties of the wax contained therein depends upon the refining history of the oil, upon whether the oil is a residuum or a distillate, and upon whether the oil has been freed or partially freed from asphaltic impurities.

I have found that certain residua derived by fire and steam or vacuum reduction from various paraffin or mixed type crudes and having viscosities within a certain range, when added to various distillates containing uncentrifugeable wax, in proportions determined by the viscosities of the residuals and of the distillates, have the specific property of so modifying the physical state of the wax in said distillates as to render the same centrifugeable.

I have found that these residua are usually satisfactory in admixture with distillates to form dewaxing stocks only when a sufficient fraction has been distilled off to leave the residua substantially free from fractions of viscosity lower than about 100 seconds (S. U. V.) at 100° F. This means that in addition to the removal from a crude petroleum of gasoline, kerosene, and gas oil, it is necessary also to distill off a light lubricating oil which is capable of being dewaxed by pressing and is usually, therefore, designated as pressable distillate. Residua obtained in this manner from crude petroleum relatively high in asphaltic impurities are required in somewhat smaller proportions in dewaxing stocks than are residua derived from crude petroleum relatively low in asphaltic impurities. The usual sulfuric acid refining process, however, tends to destroy this wax modifying property. It is of obvious advantage, therefore, in the manufacture of the blended stocks of particularly low pour test according to the present invention, to use residua requiring a minimum of refining prior to dewaxing; that is the residua are advantageously derived from high grade crudes.

By "high grade" crude is meant a wax-bearing crude having a sufficiently small content of asphaltic material to enable the use of residuals derived therefrom as lubricating stocks without

drastic refining. As examples of such crudes there may be mentioned any "Pennsylvania grade" crude oil and Oklahoma crudes from Burbank, Tonkawa, Glenn Pool and Oklahoma City. In addition to these oils, some restricted fields in Texas produce oils of high grade "Mid-Continent quality" that would be useful in the process herein described.

I have found that distillates which can be employed in any considerable proportions with residua, prepared as above described, should have viscosities, as a whole higher than about 50 seconds (S. U. V.) at 210° F. and should contain substantially no fraction having a viscosity lower than 100 seconds (S. U. V.) at 100° F. Distillates which have not been treated with sulfuric acid require less residua in the mixed dewaxing stock than do distillates freed or partially freed from asphaltic and other similar impurities by such refining, unless the dewaxing stock is acid treated prior to dewaxing.

I have found that the amount of residuum required to be added to a given distillate in order to produce a satisfactory dewaxing stock, in accordance with my invention, is a function of the viscosity of each constituent. A determination of the proper proportion of residuum to add often cannot be based on the viscosity of the mixture, since the lighter fractions contained in a residuum have no substantial effect in rendering wax centrifugeable and may be detrimental. The fractions having the higher viscosities are most effective and these fractions are the only ones which need to be considered in estimating the proper proportion of residuum in a dewaxing stock.

The residua from various crudes which have been reduced without cracking until the viscosity of the stream is not lower than about 100 seconds (S. U. V.) at 100° F. will have various viscosities depending upon the type of crude reduced and the degree of fractionation during reduction. While such residua are usually effective in modifying wax to render the same centrifugeable, the reduction may be carried on, if desired, to obtain higher viscosity bottoms which may be used to somewhat greater effect in admixture with suitable distillates as described. For example, in many of my experiments, I have reduced a selected north Oklahoma crude until the viscosity of the stream was about 150 seconds (S. U. V.) at 100° F. to obtain a residuum with a typical viscosity of 190 seconds (S. U. V.) at 210° F. Such a residuum is highly satisfactory for my process.

In general it may be said that when a high grade crude has been reduced until the viscosity of the overhead stream is at least 100 seconds (S. U. V.) at 100° F. and when the viscosity of the resulting residuum is from about 100 to 250 seconds (S. U. V.) at 210° F., such residuum will act as an effective wax modifier. Most high grade crudes when subjected to such a reduction will produce a residuum having a viscosity of roughly 175 seconds (S. U. V.) at 210° F., as a typical or average value. I have found that the amount of such a residuum required to be added to a given distillate in order to modify its wax content can be considered as bearing a rather definite relationship to the viscosity of said distillate conforming with certain formulæ given below, which formulæ can be employed for purposes of calculating the percentage of residuum required in a given dewaxing stock containing distillates.

These formulæ are as follows:

Optimum requirements

$$R = 85 - 5.6\sqrt{V - 50}$$

5 Minimum requirements

$$R = 65 - 5.6\sqrt{V - 50}$$

wherein R represents the percentage of the residuum required in the mixture, and V represents the viscosity of the distillates.

If the high grade crude has not been reduced to the proper extent the above formulæ can still be employed with suitable correction for the proportion of residuum in the mixture having a viscosity of above 175 seconds (S. U. V.) at 210° F., for example. The lighter ends in a residuum which has not been reduced to the extent noted act as an unpressable distillate and at times allowance must be made for such ends in estimating the amount of residuum required. The proportion of residuum having a viscosity of above 175 seconds (S. U. V.) at 210° F. may be considered the "effective residue" in wax modification, for purposes of this calculation.

The above formulæ are, of course, purely empirical but are based upon the results of a large number of experiments. These formulæ appear to be applicable to a wide variety of residua from high grade crudes. It is convenient in conducting my process to prepare the wax modifying residual by batch reduction until the stream shows a viscosity of about 150 at 100° F. Usually in this case no correction for the light ends in the residuum need be made. In any case it is relatively simple to determine the amount of "effective residue" in a residuum by a further laboratory reduction to the desired point.

I have found that the desirable and feasible range of viscosities of residua for wax modification from paraffin or mixed type crudes is from 100 to 250 seconds (S. U. V.) at 210° F. In obtaining such residua by batch reduction the viscosity (S. U. V.) of the stream may be brought up to from 100 to 1000 seconds (S. U. V.) at 100° F., or an equivalent degree of reduction for continuous tube still reduction may be employed.

I have found it advantageous to employ crudes having relatively low asphaltic content as a source of the residua to be employed in my process. The higher grade paraffin base crudes from the north Oklahoma fields are typical. If desired, the "bloom" of the finished bright stock to be obtained in my process may be improved at the same time by using, as a residual, the synthetic green bloom agent described and claimed in my copending application, Ser. No. 381,398, filed July 26, 1929, now Patent No. 1,863,473. The residual described in that application is derived from a naphthenic type crude which is reduced in the presence of anhydrous aluminum chloride thereby producing a highly potent green bloom agent.

My invention is illustrated and can be more readily explained by referring to the accompanying drawing in which the figure represents a flow chart of two embodiments of my invention. The treatment of two types of oils is illustrated in this figure. Crude oil A represents a crude containing sufficient asphaltic material to render the use of residuum as dewaxing stock uneconomical. This type may be represented, for example, by the crude produced in the east Texas field, containing unusually large quantities of wax and hence presenting a special problem, or it may be represented by the Seminole type Oklahoma crude, containing lower and more nearly nor-

mal quantities of paraffin wax. Crude oil B represents a crude containing minimum amounts of asphaltic material, for example, a selected high grade north Oklahoma crude of paraffinic type. The residuum from the type B oil is used to modify the wax structure in the heavy distillates of the type A.

Referring to the upper left hand corner of the figure, the illustrated crude A may represent a Seminole type Oklahoma crude, for example, while crude B, at the upper right side may represent a special north Oklahoma crude. In the specific embodiment shown in this figure the total crude charge (100 per cent) is made up of an 80 per cent charge of the Seminole crude and a 20 per cent charge of the north Oklahoma crude. The percentage yields indicated in this figure are based on the total crude charge. The percentage yields based on the individual charges may be found from these figures by dividing by 0.8 and 0.2, respectively, for the Seminole and for the north Oklahoma crudes.

In the figure crude A is shown being fed into a still where it is subjected to atmospheric fire and steam distillation. The outlet temperature of this still is maintained at a temperature of about 775° F. Gasoline, kerosene, gas oil and a pressable distillate are distilled off, the subsequent treatment of these products forming no part of the present invention. About 4 per cent by volume of the total charge (i. e. 5 per cent of the Seminole crude) is recovered as an unpressable distillate. About 15 to 17 per cent (or 18 to 20 per cent of the Seminole crude) is obtained as bottoms and these bottoms are sent to a vacuum still. In this vacuum operation, the bottoms are subjected to a further reduction with fire and steam under high vacuum. In some cases I employ a vacuum still for this secondary reduction which, as a matter of operating economy and convenience, is not equipped with a fractionating tower. In other cases the vacuum still is equipped with a fractionating tower for separation of any lighter fractions from the heavy overheads. In using a vacuum still that is not equipped with an efficient fractionating tower, a small amount of a lighter unpressable distillate fraction is usually separated by partial condensation in a 2-stage condenser. This unpressable distillate is a material of such wide cut as to render it at times difficult to process for wax removal. It is convenient, therefore, to return this wide cut fraction, as shown, to the atmospheric still for recycling with the crude charge so as to subject it to more effective fractionation in the efficient fractionating tower of the atmospheric still. The final result of this recycling is that a close cut unpressable distillate is obtained from the fractionating tower of the atmospheric still. Overhead cylinder stock is obtained from the vacuum still and represents about 6 per cent of the original charge (i. e. 8 per cent of the Seminole crude). This is advantageously mixed with the unpressable distillate from the same crude, obtained from the atmospheric still.

Crude oil B, a source of supply of which is illustrated in the upper right hand side of the figure, represents a special north Oklahoma crude, for example. As shown, this may be fed to an atmospheric still equipped with an efficient fractionating tower, and there subjected to fire and steam reduction, the outlet temperature being held at approximately 725° F. Gasoline, kerosene, gas oil and a pressable distillate are fractionated off and

recovered. An unpressable distillate, representing about 1 per cent by volume of the total charge (i. e. 3 to 5 per cent of this crude) is obtained and may be mixed with the distillates from crude

- 5 A. Crude B is reduced to a residuum having a viscosity of about 190 seconds (S. U. V.) at 210° F., or to a residuum of viscosity of the order of that secured by batch reduction of the crude until the viscosity of the stream is about 150 seconds (S. U. V.) at 100° F. This residuum represents about 5 per cent by volume of the total charge or about 25 per cent of crude B. This residuum constitutes the wax modifier, and is mixed with the above described unpressable distillates and overhead cylinder stock to modify the wax separating qualities of those materials. The entire above described mixture of unpressable distillate and overhead cylinder stock and residue has a viscosity of about 132 seconds (S. U. V.) at 210° F., the residue from crude B forming about 33 per cent by volume of the total charge and having a viscosity of about 190 seconds (S. U. V.) at 210° F.

- 25 The mixture of residue and distillates is portioned according to the before-mentioned empirical formula and wax separation can then be accomplished by well-known centrifugal methods. It was found in the present specific embodiment that a minimum of about 25 per cent of residue was required to be added to the overheads to produce a centrifugeable mixture. I generally prefer, however, to add an additional 5 to 10 per cent or so to be on the safe side, as well as to make the mixture somewhat more readily centrifugeable. In the present instance, therefore, I mixed the bottoms and overheads in the ratio of 33:67 as an optimum proportion.

- The mixture of heavy distillates and residue, as shown and described, is then refined by methods, many steps of which have been long known in the art and which form no part of the present invention. In one way of working, by opening valves C and D and closing valves E, F, G and H, the blend of overheads and residuals may, as shown, be diluted with about 11 per cent by volume of heavy uncracked kerosene distillate. A heavy kerosene distillate is advantageous for this dilution as giving the maximum reduction in viscosity with but small loss by evaporation during refining. The dilution renders the blend more suitable for acid treatment in lowering the viscosity so as to promote more ready agglomeration and settling of acid sludge particles.

- Acid treatment is carried out in agitators. 55 Sulfuric acid, preferably of around 98 per cent, in the proportion of 38-50 pounds per barrel of oil may be employed. The acid treatment may be carried out in two steps, if desired. Starting temperatures of about 110-115° F. are employed for summer conditions and 120-125° F. in the winter, the temperature of the batch usually rising about 20° F. during treatment. Substantially all or a considerable portion of the acid sludge may be removed in the agitators, the rest being separated in air blown settling pans at temperatures ranging from 120 to 150° F., as illustrated in the figure.

- The sour oil from the acid treatment is neutralized with fuller's earth. This is added in the proportion of about 0.5 pound per gallon of sour oil and the mixture is then subjected to a combined digestion and reduction step in a continuous tube still and tank digester system. The temperature in this operation is raised in

progressive steps by recycling in the tube heaters until a temperature of about 550° F. is reached. The kerosene distills off during this step. A small amount of steam is passed through the digester drums which prevents discoloration of the oil during treatment and assists in removal of the kerosene. The kerosene-freed mixture of oil, still containing fuller's earth, is then sent through a pressure filter at a temperature of about 280° F. for removal of the fuller's earth. The product is now ready for wax removal.

In order to remove wax the oil is first cut with 60-70 per cent (based on the diluted mixture) of 60 gravity naphtha, the oil-naphtha mixture at this stage usually testing from 45 to 47 degrees A. P. I. This mixture is chilled in batch or continuous chillers which usually reduce the temperature to from -25° to -50° F. according to the pour test desired in the final product. The average rate of chilling in this step should be slow, say from 2° to 5° F. per hour. The mixture is then centrifuged for removal of the wax. It is at this point that the effect of the viscosity composition characteristics of the dewaxing stock is made evident. The wax from the mixture prepared in accordance with my invention separates cleanly and flows readily from the centrifuge, and the dewaxed mixture yields a product of satisfactorily low pour test.

After wax removal the mixed oil and naphtha is sent to a still in which a fire and steam reduction is effected. This still is advantageously of the continuous type equipped with high efficiency, bubble type fractionating towers, operating with a still outlet temperature of around 725° F. The naphtha is recovered overhead and an overhead neutral oil is also obtained by taking a special cut. The pour test of the overhead neutral lubricating oil obtained from this still is close to that of the residuum, when satisfactory centrifugal dewaxing has been effected as a result of the proper control of operating conditions. The residuum from the still is finally passed through fuller's earth or clay filters, taking filter cuts of the required color; or is refined to the desired color by contacting with fine decolorizing clay, or other color adsorbing material. The final blended lubricant obtained by the above procedure may have the following typical tests:

Gravity, A. P. I.	23.0
Vis./210° F.	153
Flash, P. M. ° F.	525
Flash, O. C. ° F.	550
Fire, O. C.: ° F.	640
Pour, ° F.	+5
Color, N. P. A.	7.5
Carbon residue: %	1.91

In producing the above product a temperature of about -40° F. is required in the chillers. If market demands call for a lower or higher pour test this can be readily obtained by a suitable change in the temperature used in the chilling step. When the temperature is lowered an allowance must be made for the increased viscosity of the dewaxing stock, by slightly increasing the proportion of naphtha used in dilution. Otherwise a satisfactory rate of subsidence of the wax particles in the centrifuge will often not be obtained. For example, in chilling to a temperature of -50° F., the proportion of naphtha must be increased about 5 per cent over that required in chilling to -40° F. The pour test of the product will usually be lowered from 5° to 7° F.

by this procedure. Roughly it requires a reduction of from 1.5° to 2° F. in the chilling temperature of the oil-naphtha mixture to produce a lowering of 1° F. in the pour test of the product.

The above process is capable of variation to suit the particular type of crude oil A which is being treated. Probably the most difficult of such oils to handle are those containing large quantities of wax. A typical example of such a crude oil is that derived from the east Texas field. This particular crude, which contains more than double the usual amount of wax, requires a modification of the above procedure for most economical processing. A double dewaxing operation may be employed to advantage, as the amount of wax to be removed is so great that a single dewaxing is more costly due to the large amount of refined lubricating oil that is lost mechanically in the wax. Such a double dewaxing operation may be accomplished as shown in the figure by closing valves C and D and opening valves E, F, G and H.

As illustrated the contents of the mixer, which contains unpressable distillate and overhead cylinder stock from crude A and unpressable distillate and bottoms from crude B, as described above, may first be cut with naphtha to the amount of about 55-65 per cent by volume of the mixture. This mixture may then be run through chillers to reduce the temperature to say -10° F. and then through a centrifuge to remove the larger portion of the wax. The mixture may then be reduced to recover the naphtha. The reduced oil may then be mixed with about 11 per cent by volume of kerosene, to which mixture may be added, if required, additional bottoms, or modifying residuum, from the crude B atmospheric still. The resulting mixture may then be acid treated, clay contacted, mixed with naphtha and dewaxed for a second time at lower temperature following the procedure outlined previously. The first dewaxing in this procedure is to remove the excess of wax contained in such oils and to produce an oil of more nearly normal wax content which can then be processed in the usual manner, without excessive loss of valuable refined lubricating oil with the separated wax. In this example of centrifuging, before and after acid treatment, the same rules apply for proportioning in accordance with viscosity.

The wax-oil mixture discharged from the centrifuge in the secondary dewaxing operation by the above-described procedure usually contains a large proportion of lubricating oil. This wax-oil mixture may be recycled or added to the oil mixture subjected to the primary centrifuging before acid treatment, so as to effect removal of the larger portion of its wax content under controlled conditions tending to concentrate or pack it in hard condition. In this manner it is possible to return to the system the greater portion of the oil that would otherwise be lost in the wax discharge from the secondary operation on the refined mixture.

The procedures described above are capable of wide variation while producing the desired results. Thus, if the crude is a border line material containing only a small amount of asphaltic substance, a residuum from such a crude may economically be mixed with the high viscosity overheads from separate reduction of an additional quantity of the same crude to form a satisfactory dewaxing stock. In this case the crude A and crude B shown in the flow chart may both

be represented by the same oil. In this case, however, it may be advantageous to acid treat crude B before reducing it to residuum which then becomes a constituent of the dewaxing stock.

In another modification of my invention, crude A can be reduced directly to a heavy flux oil by fire and steam, completing the reduction under vacuum conditions without transferring the bottoms to a special vacuum still. In this reduction an unpressable distillate and an overhead cylinder stock may be recovered. These are treated with sulfuric acid either separately or in admixture. For example, the unpressable distillate may be acid treated, centrifuged for wax removal, etc., to finally recover a heavy neutral oil. In this case the wax removal is made possible, according to my invention, by adding a calculated amount of bottoms from crude B prior to acid treatment. The overhead cylinder stock is then usually treated in substantially the same manner to recover a bright stock. In case the unpressable distillate and overhead cylinder stock from crude A are mixed before treatment, bottoms from crude B are added to this mixture prior to acid treatment, after which the steps are the same as those indicated on the accompanying flow chart.

Several different methods may be employed for neutralizing the "sour oil" obtained after treatment with sulfuric acid. Thus, the clay contacting and reduction step described above can be replaced by a simple heat treatment in a still, with reduction but without the addition of clay. The maximum temperature used in the still is about 600° F. Clay filtering, either before or after admixture with naphtha, is usually required before chilling and centrifuging if this type of heat treatment is employed. Another method which may be employed for neutralizing the sour oil from the acid treatment is a washing with water, dilute alkali and steam. Sufficient 10 per cent caustic soda solution may be used, for example, to produce a neutral reaction. Steam may then be blown through the mixture of oil and caustic followed by drawing off the caustic solution and washing with about 20 per cent by volume of hot water with steam agitation. If desired, the sour oil can be split, part being neutralized by one method and part by another.

Various proportions of the bottoms from crude B may be employed as a wax modifier. It is generally, of course, advantageous to keep this proportion as low as possible since the supply of special crudes best suited for this purpose is limited. Some experimental work is often required to obtain optimum conditions. North Oklahoma Burbank crude is the best type of crude B with which I am now familiar, while straight Glenn Pool crudes are nearly as satisfactory. Other mid-continent crudes of paraffin base are suitable.

In order to illustrate the proportion of residuals required to produce a satisfactory dewaxing stock, the following table is included, this table being based upon results of a number of experimental runs. The table shows the proportions of overheads of various viscosities and residual oils that have been found to give satisfactory results by the centrifugal dewaxing operation when employed in the proportions noted and when the process details described in this application are followed. The overhead stocks of the viscosities indicated were derived from the lower grade Seminole type Oklahoma crude, while the modifying bottoms were derived from the high grade North

Oklahoma crude by reduction to a residuum testing around 190° vis./210° F. S. U. V. In the first line of the table are given the viscosities (at 210° F.) of the overhead stocks to be modified and in the last line there is shown the per cent by volume of the north Oklahoma bottoms required to modify the same. The values given show approximately the optimum quantities of bottoms required. The minimum amounts required are often from 5 to 10 per cent lower than the values given.

Vis. of overheads—	50	60	75	100	125	150	175	200
Percent of bottoms required—	75	55	45	35	30	25	15	10

It is evident from this table that the proportion of the residual modifying agent required rapidly increases as the viscosity of the overheads is reduced. The data contained in this table follow the results calculated from the formulae given ante with fair precision.

Other modifications of the above processes may be better adapted to suit special types of crudes. Various changes may be made in the specific steps of the processes and even the sequence of several of the steps may be altered without departing from the scope of my invention. For example, the temperatures, and pressures of the distilling steps may be varied. The proportion of kerosene employed as a diluent in the acid treating step, the concentration and proportion of acid used in this step, and the proportion of fuller's earth used, may all be altered. In some cases wax removal may be accomplished before instead of after acid treatment, etc. Such permissible variations will be evident to those skilled in the art.

In its broad aspects my invention contemplates the extraction of wax from a mixture of overheads and residuals by centrifugal force, the proportions of overheads and residuals in said mixture being controlled in relation to the viscosities of said overheads and residuals and, more specifically, said proportions being controlled in accordance with the formulae given ante. My invention is more particularly adapted to the case where the overheads and residuals are derived from different crude bases, the overheads being derived, for example from a crude containing asphaltic impurities and the residuals being derived from a crude containing appreciably less such impurities. My invention also includes the dewaxing stock formed from a mixture of such residuals and overheads, as well as the finished blended lubricants obtained therefrom.

What I claim is:

1. In the manufacture of lubricating oils, the process which comprises mixing non-centrifugeable, wax-containing overheads with residuals capable of treatment in a centrifugal and dewaxing said mixture by centrifugal force, the percentage of said residuals in said mixture being not less than that calculated from the formula

$$R=65-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

2. In the manufacture of lubricating oils, the process which comprises mixing non-centrifugeable, wax-containing overheads with residuals capable of treatment in a centrifugal and dewaxing said mixture by centrifugal force, the

percentage of said residuals in said mixture lying between the values calculated from the following formula:

$$R=85-5.6\sqrt{V-50}$$

and

$$R=65-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

3. In the manufacture of lubricating oils, the process which comprises mixing non-centrifugeable, wax-containing overheads with residuals capable of treatment in a centrifugal and dewaxing said mixture by centrifugal force, the percentage of said residuals in said mixture being approximately that calculated from the following formula:

$$R=85-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

4. In the manufacture of blended oils, the process which comprises reducing a high grade crude until the residuum as a whole has a viscosity above 100 seconds S. U. V. at 210° F. and is substantially free from fractions of viscosity lower than about 100 seconds S. U. V. at 100° F., mixing this residuum with an unpressable non-centrifugeable distillate having for the whole a viscosity higher than about 50 seconds S. U. V. at 210° F. and substantially free from fractions having a viscosity lower than 100 seconds S. U. V. at 100° F., in controlled portions to produce a dewaxing stock capable of being centrifuged and then dewaxing said mixture by centrifugal force, the percentage of said residuals in said mixture being not less than that calculated from the formula

$$R=65-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

5. In the manufacture of blended oils, the process which comprises reducing a high grade crude until the residuum as a whole has a viscosity above 100 seconds S. U. V. at 200° F. and is substantially free from fractions of viscosity lower than about 100 seconds S. U. V. at 100° F., mixing this residuum with an unpressable non-centrifugeable distillate having for the whole a viscosity higher than about 50 seconds S. U. V. at 210° F. and substantially free from fractions having a viscosity lower than 100 seconds S. U. V. at 100° F., in controlled proportions to produce a dewaxing stock capable of being centrifuged and then dewaxing said mixture by centrifugal force, the percentage of said residuals in said mixture lying between the values calculated from the following formula:

$$R=85-5.6\sqrt{V-50}$$

and

$$R=65-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

6. In the manufacture of blended oils, the process which comprises reducing a high grade crude until the residuum as a whole has a vis-

costly above 100 seconds S. U. V. at 210° F. and is substantially free from fractions of viscosity lower than about 100 seconds S. U. V. at 100° F., mixing this residuum with an unpressable non-centrifugeable distillate having for the whole a viscosity higher than about 50 seconds S. U. V. at 210° F. and substantially free from fractions having a viscosity lower than 100 seconds S. U. V. at 100° F. in controlled proportions to produce a dewaxing stock capable of being centrifuged

and then dewaxing said mixture by centrifugal force, the percentage of said residuals in said mixture being approximately that calculated from the following formula:

$$R=85-5.6\sqrt{V-50}$$

wherein R represents the per cent of residuals in said mixture and V represents the viscosity of the overheads.

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