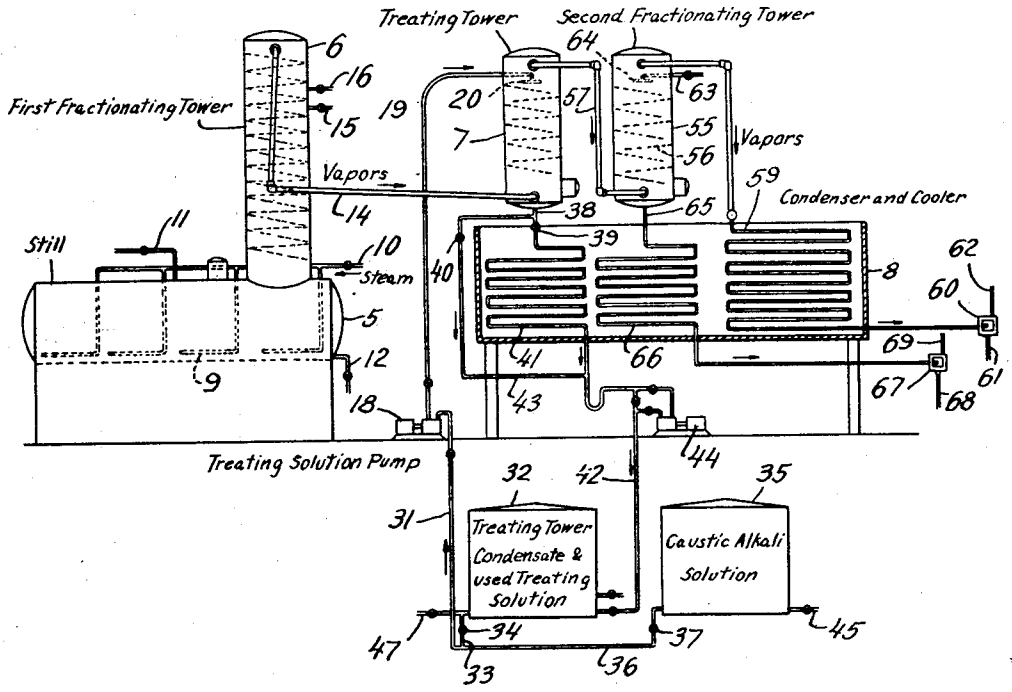
March 11, 1930. G. H. TABER, JR 1,749,896

MANUFACTURE OF LUBRICATING OIL

Original Filed Jan. 14, 1925



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Patented Mar. 11, 1930

1,749,896

UNITED STATES PATENT OFFICE

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MANUFACTURE OF LUBRICATING OIL

Original application filed January 14, 1925, Serial No. 2,262. Divided and this application filed May 26, 1928. Serial No. 280,790.

This invention relates to improvements in the distillation and refining of lubricating oils. The invention provides an improved method of producing lubricating oils from oils and distillates containing lubricating oil fractions, such as crude petroleum oil, topped crude oil, and crude lubricating oil distillates. The invention includes an improved combined distilling and refining operation, improvements in the fractionation of oils and distillates containing lubricating oil for the separation of the desired lubricating oil fraction free from heavier and lighter components, an improved method of refining lubricating oils, and improvements in continuous distillation processes for the production of lubricating oils. This application is a division of an application filed January 14, 1925, Serial No. 2,262.

Hitherto, in the methods generally employed for the production of lubricating oils from crude oil, the crude oil has been initially subjected to a fractional distillation, either by batch operations or in a series of continuous stills, and a broad lubricating oil distillate fraction thereby separated from the crude oil; and this lubricating oil fraction has then been subjected to redistillation in the presence of caustic alkali introduced directly into the body of oil undergoing distillation for the production of refined lubricating oil distillates, the caustic alkali solution being employed to remove undesirable constituents such as naphthenic acids and asphaltic compounds which if allowed to distill over the finished lubricating oil distillates would render them bad colored and difficult to handle in further treatment. It has been proposed to apply the caustic alkali treatment directly to the crude oil undergoing distillation during the time in which the broad lubricating oil fraction was being produced, but such proposals have not been successful principally because of excessive deposition within the still due to the accumulation of caustic alkali in the residuum and to the combined effect of such caustic alkali and impurities normally present in the crude oil.

The process of the present invention, as applied to the production of lubricating oils, comprises continuous vaporization of the lubricating oil fractions from the crude lubricating oil-containing oil or distillate, treatment of the vaporized lubricating oil constituents with a caustic alkali, and subsequent condensation of the treated lubricating oil constituents.

According to the present invention, the vaporized lubricating oil constituents are subjected to treatment with caustic alkali while in the vapor phase. The alkali treatment of the lubricating oil fraction or fractions is thus effected before the lubricating oil constituents are condensed and in this way redistillation for treatment of the lubricating oils with caustic alkali is eliminated. Moreover, the refining operation, when carried out in this way, has several other important advantages. Thorough and intimate contact between the caustic alkali and the lubricating oil is effected and the refining action advantageously promoted. Likewise, the entire lubricating oil distillate fraction is positively subjected to thorough treatment with the caustic alkali; in the ordinary distillation with alkali the initially distilled components may escape without thorough contact with the alkali. The caustic alkali treatment of the present invention is also combined advantageously with subsequent fractionating operations for separation of the desired lubricating oil fractions. The elimination of a distilling operation, particularly the crude distillation as ordinarily carried out, also assists in eliminating cracked products from the finished lubricating oil products. Cracked constituents are usually characterized by low viscosity as compared to their boiling point and are accordingly usually objectionable in lubricating oils of corresponding boiling range. Other advantages of the invention will be apparent from the following description.

In carrying out the present invention, the lubricating oil fraction may be vaporized from the crude oil or crude lubricating oil distillate in a vaporizing still and this vaporization may be effected under atmospheric pressure or it may be effected under a subatmospheric pressure. The vaporization may

be carried out with or without the introduction of steam or an inert gas. One advantageous way to carry out the vaporization is with the introduction of steam and under reduced pressure. After vaporization, the vaporized lubricating oil constituents may be subjected to a fractionating operation, for example, by passing the vapors from the vaporizing still through a fractionating tower, or the operation of the vaporizing still may be controlled so that only the desired vapors of the desired lubricating oil fraction are produced. Where a fractionating tower is employed, it may be externally cooled or it may be cooled by introduction of a refluxing agent such as a portion of the distillate produced in the operation; and the fractionating tower may be arranged to discharge reflux back into the still or the heavier constituents separated in the tower may be removed from the tower without being returned to the still. Where a fractionating tower is employed in connection with the vaporizing still, the crude oil or crude lubricating oil distillate may be introduced into the tower and employed as a refluxing agent therein; and the operation of the tower and vaporizing still may be controlled so that vaporization of the desired lubricating oil fraction from the crude material introduced into the tower is substantially completed in the tower.

From the vaporizing still, or from the fractionating tower connected with the vaporizing still, the vaporized lubricating oil constituents are conducted to a treating tower in which they are subjected to treatment with caustic alkali in accordance with the present invention. In the treating tower, the lubricating oil vapors are thoroughly and intimately contacted with a caustic alkali solution. The treating tower may be of conventional baffle tower construction arranged for introduction of the vapors into the lower part of the tower and of the treating solution into the upper part of the tower so that the lubricating oil vapors are contacted in countercurrent flow with the caustic alkali solution. In the treating tower, a part of the oil vapors are condensed and the condensate mingles with the used treating solution. The caustic alkali content of the condensate mixture may be employed for recirculation through the tower for further treatment of the lubricating oil vapors passing therethrough.

The caustic alkali solution employed may, for example, be an aqueous solution of sodium hydroxide of a density of between about 10° and 20° Bé. The minimum amount of caustic alkali solution employed should be at least sufficient to remove the undesirable constituents from the lubricating oil vapors being treated, but the maximum amount employed may vary widely as will appear hereinafter. For example, the rate of introduction into the treating tower of the sodium hydroxide solutions just referred to may vary from ½% to 15% or more on the weight of vapors entering the treating tower. An excess of the caustic alkali solution, over that required to effect the desired refining operation, does not injure the quality of the lubricating oil distillate fractions produced, but such an excess of treating solution may produce an increased cooling effect in the tower with the condensation of an increased proportion of the vapors therein. Condensation within the treating tower may be regulated by controlling the amount of treating solution introduced therein. Where an aqueous solution of caustic alkali is employed in the treating tower, part of the water may be evaporated from the solution in the tower with resulting increase in concentration of caustic alkali in the solution, and the condensate mixture, including the used caustic alkali solution and any condensate produced in the treating tower, which accumulates may comprise a suspension of caustic alkali in oil in admixture with a more or less concentrated solution of caustic alkali. This mixture, with such additions of fresh caustic alkali solution as are necessary to replace alkali consumed in the treating tower, may be recycled through the tower for further treatment of the lubricating oil vapors passing therethrough. Before being recycled through the tower, a separation may be effected between the oil component of the mixture and the caustic alkali-containing component, and the separated caustic alkali containing component of the mixture returned to the treating tower. After discharge from the treating tower, the condensate mixture may be cooled and the oil component separated from the used caustic alkali solution, for example by decantation after allowing the caustic alkali solution to settle.

The operation of the treating tower is regulated so that the main portion of the vaporized lubricating oil constituents pass therethrough without being condensed therein. For carrying out the operation in this way, the treating tower is thoroughly lagged to prevent heat loss and the treating solution may be preheated before introduction into the treating tower. For example, the condensate mixture from the treating tower, or the alkali containing component of the condensate mixture, may be admixed with fresh alkali solution without intermediate cooling and the mixture directly reintroduced into the tower. In this manner, the treating tower operation can be controlled so that only a minimum proportion of heavier constituents is condensed therein. Any lubricating oil constituents condensed in the treating tower may be separated from the tower condensate mixture and recovered.

Before the vapors escaping from the treating tower are finally condensed, they are sub-

jected to a fractionating operation. They may, for example, be passed through a fractionating tower in which the desired lubricating oil fractions are condensed and through which the constituents lighter than those desired in the lubricating oil fractions are allowed to pass uncondensed. This fractionating tower is employed in conjunction with the treating tower for the production of a closely controlled lubricating oil distillate fraction by regulating the operation of the treating tower to condense heavier constituents and by regulating the operation of the fractionating tower to permit lighter constituents to escape uncondensed therefrom. The uncondensed vapors escaping from the fractionating tower are condensed, for example, in an ordinary water cooled condenser.

The process of the present invention may be carried out in conjunction with a series of continuously operated vaporizing stills in which a series of progressively heavier fractions are treated for the separation of progressively heavier distillates, or through which the crude oil or crude lubricating oil distillate is successively passed with the vaporization therefrom of progressively heavier fractions in successive stills of the series. Where the vapors from several such stills are passed through treating towers in which they are subjected to treatment with caustic alkali in accordance with the present invention, the oil component of the condensate mixture separated in towers operating on lighter vapors may be introduced into treating towers operating on heavier vapors with improvement of the fractionation effected in the several distillation stages. The caustic alkali associated with the oil in the condensate mixture from preceding treating towers may be introduced with the oil into successive treating towers and employed for treatment of the heavier vapors therein, or the caustic alkali component of the condensate mixture may be separated and fresh alkali supplied to successive treating towers. The separated caustic alkali mixture may be reused in the treating tower from which it is discharged.

The invention will be further described in connection with the accompanying drawings which illustrate, in a diagrammatic and conventional way, one form of apparatus adapted for the practice of the process of the invention; but it will be understood that this further description and illustration are intended for the purpose of exemplification and that the invention is not limited thereto.

The apparatus illustrated comprises a vaporizing still 5, a fractionating tower 6, a treating tower 7, another fractionating tower 55, and a cooler and condenser 8. The still illustrated is of the direct fire heated type and is provided with perforated pipes 9 for the direct introduction of steam into the body of oil in the still. The perforated pipes 9 are connected to a common header 10 which is in turn connected to a steam boiler. The still is also provided with a charging line 11 and a residue draw-off 12. The fractionating tower 6 is arranged above the still. This tower is provided with a helical baffle 13 and the vapors from the still pass upwardly through the helical passage in the tower escaping from the upper end of the tower through the vapor line 14. Connections 15 and 16 are arranged near the upper end of the tower for the introduction of charging stock into the still through the tower or for the introduction of a refluxing medium into the tower.

From the vapor line 14 the vapors enter the treating tower 7, the tower shown being of conventional baffle construction, and pass upwardly therethrough about the baffles 17 arranged in the tower. The second fractionating tower 55 may also be of conventional baffle construction being provided with baffles 56. A pump 18 is arranged to force caustic alkali solution into the upper end of the treating tower through connection 19 and the spray head 20 arranged in the upper end of the tower. The vapors escaping from the treating tower enter the lower part of the fractionating tower 55 through the vapor line 57 and pass upwardly therethrough over the baffles 56, escaping from the upper end of the fractionating tower through the vapor line 58. The vapors escaping through the vapor line 58 enter the condensing coil 59 where they are cooled and condensed and discharged into a look box 60 which is provided with an outlet 61 for condensate and a vent 62 for uncondensed vapors and gases. The fractionating tower 55 may be externally cooled or it may be cooled by the introduction of a suitable refluxing medium through connection 63 and the spray-head 64 arranged in the upper end of the tower. Where the fractionation is effected by the introduction of a refluxing medium, such as a part of the condensed distillate, it may be improved by lagging the tower to prevent variable heat loss. The condensate collecting in the lower part of the fractionating tower 55 is discharged through connection 65 and the cooling coil 66 to the look box 67 which is provided with a condensate outlet 68 and a gas vent 69. For operation under reduced pressures, the gas vents 62 and 69 and the condensate outlets 61 and 68 may be connected to receivers and vacuum pumps and condensate pumps. The treating solution is supplied to the pump 18 through connection 31, which communicates with tank 32 through connection 33 having valve 34 therein and with tank 35 through connection 36 having valve 37 therein. The condensate mixture collecting in the lower part of the treating tower 7 is discharged through con-

nection 38 to the tank 32. Connections are arranged and valves 39 and 40 are provided for discharging the condensate mixture from the treating tower to the tank 32 either through the cooling coil 41 and the connection 42 or through the by-pass connection 43 and the connection 42. The connection 42 may include a pump 44 for returning the condensate mixture to the tank 32. The tank 35 is provided for storing fresh caustic alkali solution and is provided with a charging connection 45.

The condensate mixture returned to the tank 32 from the treating tower may be recirculated through the treating tower by means of pump 18, or a separation of condensate and treating solution may be effected in the tank 32. The oil condensate is lighter than the admixed caustic alkali solution and tends to float thereon; and connection 46 is provided for withdrawing separated oil from the upper layer collecting in the tank and connection 47 for withdrawing used caustic solution from the lower layer. A separation may thus be effected in the tank 32 between the oil and the used caustic solution of the condensate mixture, and as much of the used caustic solution as may be desired may be re-employed in the treating tower together with fresh caustic solution from the tank 35.

The exposed parts of the still 5 and the fractionating tower 6 and the vapor line 14 are advantageously heat insulated or lagged to prevent unnecessary heat loss. In order to pass the maximum amount of vapor through the treating tower 7 uncondensed, the treating tower is also advantageously lagged or heat insulated.

Where the apparatus illustrated is employed for vacuum distillation, the still 5, the fractionating tower 6, the treating tower 7 and the fractionating tower 55 are suitably constructed and reinforced to prevent collapse under the excess of atmospheric pressure prevailing on the outside over the reduced pressure within the distilling apparatus, and suitable precautions are taken to prevent inleakage of air. The several vapor connections and the passages through the fractionating towers and treating tower are also made of sufficient cross-sectional area so that the desired degree of vacuum can be maintained without excessive consumption of power.

In carrying out the process of the invention in the apparatus illustrated, the still 5 is charged through the charging line 11 with oil or distillate containing the lubricating oil fractions. The still is heated and as vapors from the still rise through the fractionating tower 6 the feed of additional lubricating oil-containing charging stock is begun through connection 15. The distillation may be promoted by the introduction of live steam directly into the body of oil in the still through connection 10. The supply of additional charging stock through connection 15 may be regulated to control the refluxing operation in the fractionating tower, and this control may be supplemented by the introduction of a regulated amount of a fluxing agent through connection 16. The refluxing agent employed may, for example, be oil separated from the condensate mixture collecting in tank 32, or part of the distillates or fractions of corresponding character. The fractionating operation is regulated so that the desired lubricating oil constituents escape from the upper end of the fractionating tower 6 through the vapor line 14 and these lubricating oil vapors enter the treating tower 7. In the treating tower 7 the lubricating oil vapors are contacted in countercurrent flow with a caustic alkali solution supplied to the upper end of the tower through spray-head 20 by means of the pump 18. This caustic alkali solution may be fresh solution from the tank 35, or a solution or a suspension of caustic alkali withdrawn from the tank 32, or a mixture of the two.

The main portion of the lubricating oil vapors are passed through the treating tower with a minimum of condensation therein, and to this end the treating tower is lagged and the circulation of treating solution through the tower is maintained at the minimum necessary to effect the desired refining operation. The condensate mixture from the treating tower 7 may also be run to the tank 32 through the by-pass connection 43 without intermediate cooling, and caustic alkali solution for circulation through the treating tower made up of caustic alkali withdrawn from the heated contents of the tank 32, alone or in admixture with fresh solution from the tank 35, so that the treating solution entering the treating tower 7 through the spray-head 20 is in effect preheated.

The lubricating oil vapors escape from the treating tower through the vapor line 57 to the second fractionating tower 55 where the vapors escaping form the treating tower are subjected to fractional condensation for the separation of the desired lubricating oil distillate fraction or fractions. The condensation in the tower 55 may be effected by external cooling or by the direct introduction of a cooling medium or refluxing agent through connection 63. The condensation is controlled to condense the lubricating oil constituents without condensing the vapor constituents lighter than the desired lubricating oil constituents. In this way, a lubricating oil distillate fraction free from lighter constituents is produced directly as the condensate in tower 55. The operation of the treating tower may be controlled to separate heavier constituents, and in an operation of this

character, where heavier constituents are separated before the lubricating oil vapors enter the second fractionating tower and where lighter constituents are allowed to pass through the second fractionating tower uncondensed, a very narrow lubricating oil distillate fraction of the desired test qualities can be obtained directly, and further distillation or other treatment for refractionation or the separation of lighter constituents can be eliminated. Any lubricating oil constituents condensed in the treating tower collect in the tank 32 and may be withdrawn therefrom after separation from the caustic alkali solution therein.

The invention relates more particularly to operations in which fresh oil is supplied to the vaporizing still and residual oil withdrawn therefrom and in which a fraction of substantially uniform character is separated.

The operation may be carried out under substantially atmospheric pressure. It will be understood, however, that if the pressure in the receivers is approximately equal to atmospheric pressure the pressure in the still will be somewhat greater due to the resistance of flow of the vapors through the apparatus, but by suitably proportioning the cross sectional area of vapor passages through the apparatus to provide for free vapor flow this resistance and the consequent increase in pressure may be made substantially negligible. The still itself may be operated at atmospheric pressure, or at much lower pressures, by maintaining a suitably reduced pressure in the receivers.

The temperature at which the lubricating oil vapors are contacted with the caustic alkali solution is not critical and no definite optimum temperature can be given as this will vary with the character of the lubricating oil fraction desired and the nature of the material to be treated. In general, the higher the temperature and more intimate the contact, the smaller will be the amount of caustic alkali required. Thus the most advantageous temperature to employ in the treating operation may be determined almost entirely by the tendency of the particular oil being treated to undergo decomposition in the distilling operation to such an extent that the lubricating properties of the oil will be impaired rather than by any optimum temperature of the vapors during contact with the caustic alkali solution.

No definite optimum pressure can be given, the effect of pressure during contact of the lubricating oil vapors and caustic alkali solution being so relatively unimportant that the most advantageous pressure is determined largely by the corresponding temperature it is desired to maintain in the distilling operation to vaporize the desired fraction without impairing its lubricating properties and the almost negligible resistance of the apparatus to the flow of vapors therethrough.

The following specific examples will serve as an illustration of temperatures and pressures that may be satisfactorily employed in the manufacture of lubricating oils in the illustrated apparatus in accordance with the present invention where the distillation is effected at substantially atmospheric pressure as well as under reduced pressure.

Crude lubricating oil-containing oil may be charged to the still 5 and heated to a temperature of about 680° F. therein. Vapors generated in the still will then pass successively through the fractionating tower 6, into which fresh oil may be introduced to control the fractionating operation so as to maintain a temperature of about 650° F. at the top of the fractionating tower, and then through the treating tower 7 and second fractionating tower 55 to the condenser 59. Steam may advantageously be introduced to the still 5 through connections 9 in an amount approximately equivalent to 80 lbs. per barrel of oil charged. Under the above conditions the pressure existing in the still as indicated at the bottom of the fractionating tower may be, for example, 4 inches of mercury above atmospheric pressure due to the frictional resistance of the apparatus to the flow of the vapors therethrough. The pressure at the top of the fractionating tower 6 may be reduced by this frictional resistance to about 3 inches of mercury above atmospheric pressure. A solution of caustic alkali may be introduced into the top of the treating tower 7 at a rate corresponding to ½ lb. of caustic alkali per barrel of oil treated or slightly in excess of this amount. When operating the illustrated apparatus, as above described, the temperature of the vapors passing from the top of the treating tower 7 will be approximately 620° F. while the temperature of the liquid in the bottom of the tower will be approximately 630° F. The pressure in the treating tower due to the frictional resistance to vapor flow may vary from a pressure of about 2.8 inches of mercury at the bottom of the tower to a pressure of about 1.5 inches of mercury at the top of the tower. The pressure will continue to drop along the vapor path to about 1.3 inches of mercury at the bottom of the second fractionating tower 55 and .5 inches of mercury at the top of the second fractionating tower. Fractionation of the treated vapors in the tower 55 may be advantageously effected by maintaining a temperature of approximately 480° F. at the top of the tower. The liquid temperature at the bottom of tower 55 would, of course, be somewhat higher, for example, about 550° F.

In the treatment of less refractory lubricating oil-containing oils, the lubricating properties of which might be impaired at the temperatures employed in the distillation under

the conditions above described, the distillation may advantageously be effected at reduced pressure. For example, the still 5 may be so operated as to maintain a pressure of 60 mm. of mercury (absolute) at the bottom of the first fractionating tower 6, the pressure decreasing along the path of vapor travel to about 50 mm. of mercury (absolute) at the top of the fractionating tower; about 40 mm. of mercury (absolute) at the top of the treating tower 7; and about 35 mm. of mercury (absolute) at the top of the second fractionating tower. Steam may be introduced to the still 5 through connections 9 in an amount approximately equivalent to 20 lbs. per barrel of oil charged. The fractionating operation in the first fractionating tower 6 under such conditions may advantageously be so controlled by the introduction of fresh oil as to maintain a temperature of about 520° F. at the top of the tower. The temperature at the bottom of the first fractionating tower will then approximate 550° F. A solution of caustic alkali may be introduced to the top of the treating tower 7 at a rate sufficient to give 2 lbs. of caustic alkali per barrel of oil treated or somewhat less and the temperature of the treating tower controlled so as to maintain a temperature of 490° F. at the top of the treating tower 7. The second fractionating operation in tower 55 may to advantage be controlled so as to maintain a temperature of about 350° F. at the top of the tower when operating under the reduced pressure conditions above described.

The process of the invention is of general application to lubricating oil-containing oils and distillates. It may be applied to crude petroleums or to topped crude oils from which lighter constituents, such as gasoline, have been stripped. It is also applicable to the treatment of other oils and residues containing lubricating oil constituents, such as crude lubricating oil distillates and fractions. It may be employed in the treatment of oils of either asphaltic or paraffine base or mixed base oils. Following the distillation and refining treatment of the present invention, the lubricating oil distillate fractions may be filtered through fuller's earth or similar material or subjected to such other treatment as may be desired. Wax containing distillates, for example, may be subjected to suitable dewaxing operations in the usual manner. In referring to lubricating oils herein, reference is made to the petroleum oils characterized by oiliness and high viscosity. In general these oils may be defined as oils made up of constituents heavier than gas oil, having a gravity lower than about 35° Bé. and a viscosity upwards of about 50 at 100° F. Saybolt Universal.

I claim—

1. In the manufacture of lubricating oils the improvement which comprises continuously vaporizing lubricating oil constituents from a stock containing the same, continuously taking off the lubricating oil vapors and subjecting them as such to treatment with a caustic alkali solution, maintaining the temperature in this treatment to condense a minimum of lubricating oil constituents, continuously taking off lubricating oil vapors from this treatment free from alkali, and condensing a lubricating oil fraction from the vapors taken off from the alkali treatment, whereby said condensed lubricating oil fraction is subjected to the alkali treatment in the vapor phase but condensed out of contact with the alkali.

2. In the manufacture of lubricating oils, the improvement which comprises continuously vaporizing lubricating oil constituents from a stock containing the same, continuously taking off the lubricating oil vapors and subjecting them to a fractionating operation for condensation of heavier constituents, continuously taking off the lubricating oil vapors from the fractionating operation and subjecting them as such to treatment with a caustic alkali solution, maintaining the temperature in this treatment to condense a minimum of lubricating oil constituents, continuously taking off lubricating oil vapors from this treatment free from alkali, and condensing a lubricating oil fraction from the vapors taken off from the alkali treatment, whereby said condensed lubricating oil fraction is subjected to the alkali treatment in the vapor phase but condensed out of contact with the alkali.

In testimony whereof, I have subscribed my name.

GEORGE H. TABER, JR.

CERTIFICATE OF CORRECTION.

Patent No. 1,749,896. Granted March 11, 1930, to

GEORGE H. TABER, JR.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, line 36, after the word "over" insert the word "with"; page 4, lines 72 and 73, for the word "fluxing" read "refluxing", and line 114, for "form" read "from"; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of April, A. D. 1930.

(Seal)

M. J. Moore,
Acting Commissioner of Patents.