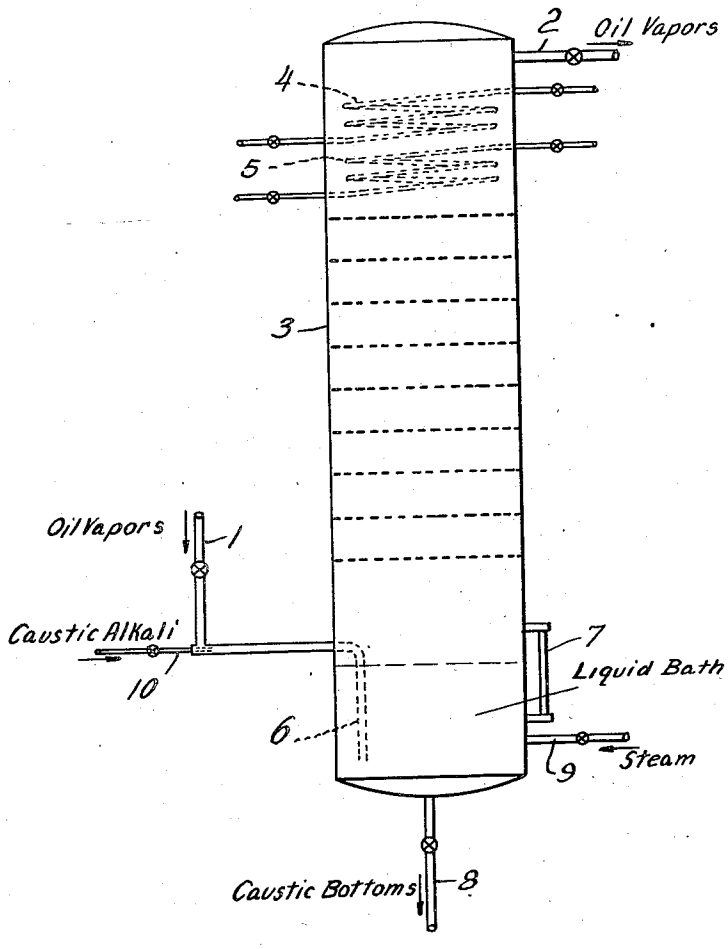
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MANUFACTURE OF LUBRICATING OILS

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INVENTORS
Eugene C. Herthel
Harry L. Pelzer
BY
Regen Hobson, Irvin & Macauley
ATTORNEYS

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EUGENE C. HERTHEL, OF CHICAGO, ILLINOIS, AND HARRY L. PELZER, OF HIGHLAND, INDIANA, ASSIGNORS TO SINCLAIR REFINING COMPANY, OF NEW YORK, N. Y., A CORPORATION OF MAINE

MANUFACTURE OF LUBRICATING OILS

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This invention relates to improvements in the manufacture of distilled lubricating oils from petroleum stocks. The invention is of special value and application in the manufacture of distilled lubricating oils from certain types of crude oils or crude oil stocks of which the Gulf coast, South Texas and the California oils may be taken as examples.

In general, lubricating oils may be defined as oils heavier than gas oil, heavier than about 35° Bé., and having viscosities upwards of about 50 at 100° F. Saybolt Universal.

There is a group of constituents, apparently characteristic of naphthene base crude oils, which are extremely objectionable as components of lubricating oils in particular. These constituents are particularly objectionable in that they render unstable lubricating oils in which they are present. They also impair the quality of lubricating oils containing them in other respects, color for example.

As yet, this group of objectionable constituents has not been definitely identified. These objectionable constituents are themselves apparently of relatively low boiling point, yet they polymerize quite readily to form a tar or tarry substance. This polymerization is promoted, for example, by the presence of air or steam. With certain types of stocks, additional quantities of these objectionable constituents seem to be formed as long as the oil is maintained at elevated temperature, this effect increasing with temperature increase.

A number of schemes of procedure have been proposed intended to avoid the objectionable incidents of the presence of this group of objectionable constituents in lubricating oils, but these proposals have not been entirely satisfactory; in some cases because of operating difficulties and in other cases because of failure to reach the real difficulty.

The present invention provides particularly an improved method of separating constituents of the objectionable group noted above from lubricating oil products produced from stocks containing these objectionable constituents, and the present invention is also useful in other applications, as will appear, in providing an improved method of treating lubricating oil products with caustic alkali.

According to the present invention, the petroleum stock containing the lubricating oil constituents is subjected to distillation and during the distillation the vapors forming the lubricating oil products when condensed are bubbled through a hot alkaline liquid oil bath in which the separated objectionable constituents are collected and from which they are discharged, the vapors after passage through the hot alkaline liquid oil bath are subjected to a refluxing operation from which reflux is returned to the bath, the temperature of the bath and the refluxing operation are controlled to maintain uncondensed the constituents forming the desired lubricating oil products, and the vapors including the lubricating oil constituents escaping from the refluxing operation are condensed to form the distilled lubricating oil products. The constituents of the raw stock suitable as components of the desired lubricating oil products are vaporized from the raw stock and these vapors are bubbled through the hot alkaline liquid oil bath by introduction beneath the surface thereof. The oil bath is maintained alkaline as the operation proceeds by supplying a caustic alkali thereto. The condensed lubricating oil products are not permitted to contact with the oily or tarry material forming the treating bath.

In the hot alkaline liquid oil bath, the lubricating oil constituents are subjected to intimate contact with caustic alkali. The caustic alkali neutralizes acidic constituents of the oil vapors and, apparently, also acts as a polymerizing agent promoting the polymerization of constituents of the objectionable group noted above. Steam may with advantage be supplied to the treating bath as a distillation medium, and when so supplied also assists in this polymerization and in some cases also seems to make the caustic alkali more active. In the refluxing operation, high boiling constituents not suitable as components of the lubricating oil products including reaction products of the hot alkaline treatment and the products of polymerization of any objectionable constituents pres-

ent are condensed and returned to the treating bath from which they are discharged.

The vaporization of the raw petroleum stock may be effected in a shell still or a pipe still, for example. The vaporization may be carried out under pressures approximating atmospheric pressure or under sub-atmospheric pressure and with or without the introduction of steam or other hot gaseous medium reducing the partial pressure of the oil vapors. As applied to crude oils, the vapors subjected to the alkaline treatment of the invention may comprise the total vaporized fraction of the crude oil.

The treating bath is with advantage externally unheated. The formation of separating carbonaceous material in the bath may thus be avoided or minimized. Contact between the alkaline material of the bath and surfaces carrying a burden of heat transfer is also thus avoided. The hot oil bath is advantageously maintained of substantially constant volume. The volume of the bath may be controlled by regulating the temperature of the caustic alkali supplied thereto, by the introduction of steam or some heated gas, by the regulated discharge of liquid from the bath, or by refluxing to the bath heavier components of the vapors taken off therefrom. The caustic alkali is advantageously supplied to the treating bath by introduction with the vapors from the vaporizing operation and in amount sufficient to maintain that excess of alkali in the bath required to neutralize completely the escaping vapors.

The vapors taken off from the treatment of the invention may be condensed or fractionally condensed in any convenient apparatus. These vapors may, for example, be passed through a series of towers in which progressively lighter fractions are condensed. As applied to crude oils, the vapors taken off from the hot oil bath may thus be condensed as a series of distillates, the heavier and more viscous fractions of the series constituting the lubricating oil products.

The invention will be further described in connection with the accompanying drawing which illustrates, diagrammatically and conventionally, one form of apparatus adapted, in conjunction with a suitable still and a suitable fractionating or condensing system, for carrying out the invention. It will be understood that the invention can be carried out in other and different forms of apparatus; this particular form is illustrated merely for purposes of exemplification. Any usual form of still and any usual fractionating or condensing system may be used in carrying out the invention and this part of the apparatus is, therefore, not illustrated.

Referring to the drawing, in the apparatus illustrated the vapors from the still are supplied through line 1 and are taken off, following the treatment of the invention, through line 2 to the fractionating or condensing apparatus, the vapors passing from line 1 through tower 3 to line 2. The tower 3 illustrated comprises three sections, a lower treating section in which the hot alkaline oil bath is maintained, a middle refluxing section, and an upper dephlegmating section. Cooling coils 4 and 5 are provided in the dephlegmating section. Baffles or trays for promoting vapor-liquid contact and heat exchange are provided in the refluxing section. A normally submerged inlet pipe 6 is provided in the treating section, connected to line 1, for the introduction of the vapors from the still beneath the normal liquid level therein. Gauge 7 is provided for observation of the liquid level. Line 8 is provided for discharge of liquid from the lower end of the tower. Line 9 is provided for introduction of steam or other hot gaseous medium into the lower end of the tower beneath the normal liquid level therein. Line 10 is provided for introduction of caustic alkali with the vapors from line 1 through pipe 6.

In carrying out the invention in the apparatus illustrated, the lubricating oil vapors supplied through line 1 are bubbled though a hot alkaline liquid oil bath maintained in the lower section of tower 3, being introduced beneath the surface thereof through pipe 6, this hot oil bath is maintained alkaline by supplying caustic alkali thereto in the form of an aqueous solution through line 10 and pipe 6, the vapors after passing through the treating bath are subjected to a controlled refluxing operation as they flow upwardly through tower 3, and the lubricating oil vapors are taken off through line 2 and separately condensed. Components of the resulting condensate or condensates contact with the treating bath in the lower section of tower 3 only as vapors; the condensate or condensates are not permitted to contact with the oily or tarry material forming this bath as such.

The following example will illustrate an operation embodying the invention, as carried out in apparatus of the type illustrated: Gulf coast crude oil having an acid number of 1.85 is supplied to a small pipe still at a rate of about 400 gallons per hour and discharged from the pipe still at a temperature of about 800° F. After separation of unvaporized residual oil, the vaporized part of the stock is supplied through line 1 to the lower end of tower 3. An aqueous solution of caustic soda, 25° Bé., is also supplied through line 10 to the lower end of tower 3 at a rate of about 5.5 gallons per hour. The temperature at the top of tower 3 is maintained in the neighborhood of 670–690° F. and the temperature of the liquid bath in the lower end of tower 3 in the neighborhood of 690–700° F. Steam is supplied through line 9 and a cooling medium is circulated through

coil 4 or coil 5 or both coils as required to maintain these temperatures. The vapors escaping through line 2 from the upper end of tower 3 are fractionally condensed to form a heavy lubricating oil having an acid number of 0.042, a light lubricating oil having an acid number of 0.014, and a composite gas oil and naphtha having an acid number of 0.014. A liquid bath of substantially constant volume is maintained in the lower end of the tower by regulated discharge of liquid through line 8.

The tower 3, particularly the lower end, is with advantage lagged or thermally insulated and the liquid bath therein heated by the introduced oil vapors. Additional heat may be supplied by the introduction of steam through line 9, steam superheated for example to 800–900° F., or by the introduction of other hot gaseous media through line 9. The temperature of the caustic alkali as introduced through line 10 may also be regulated to assist in controlling the heat balance in the tower. The hot oil bath in the lower end of the tower 3 is maintained at a temperature high enough to prevent the retention therein, condensed, of any constituents suitable as components of the desired products.

The hot oil bath in the lower end of tower 3 is with advantage maintained of substantially constant volume as the operation proceeds. Any tendency of the bath to decrease in volume may be counteracted by supplying the caustic alkali at a lower temperature, by cooling or increasing the cooling of the vapors in the upper end of the tower, by decreasing the rate of liquid discharge, or by decreasing the rate at which steam is supplied to the lower end of the tower if steam is used. Any tendency of the bath to increase in volume may be counteracted by supplying the caustic alkali at a higher temperature, by decreasing the vapors in the upper end of the tower, by increasing the rate of liquid discharge, or by supplying steam or increasing the supply of steam to the lower end of the tower.

Oil to be supplied to the vaporizing operation may be used as a cooling medium in the cooling coils 4 and 5, or some suitable external cooling medium may be so used. The cooling may be controlled by regulating the rate of circulation of the cooling medium through the coils 4 and 5.

By introducing the caustic alkali into the vapors from the still before the latter enters the hot alkaline oil bath and then introducing the mixture into the body of the liquid bath as in the apparatus illustrated, a particularly effective contact between the oil vapors and the caustic alkali is obtained. The fresh caustic alkali flows through pipe 6 in admixture with the vapors supplied through line 1 and the vapors are then bubbled through the liquid alkaline oil bath to which additional caustic alkali is continuously being added.

The invention is particularly adapted to continuous operation and the control afforded by the invention is of special value in maintaining uniform the distillate products over long periods of operation. The invention provides a particularly effective contact between the lubricating oil vapors and the caustic alkali treating agent, and in this respect also has the advantage of economy in consumption of caustic alkali. By separation of residual unvaporized oil from the vaporized components including the lubricating oil before the latter are supplied to the treatment of the invention, the caustic tar or caustic residue produced may be kept separate from the major part of the residue and may be kept in but a minimum proportion of the total residue produced in the complete operation. Further, in the operation of the invention, difficulties due to entrainment of reaction products of the alkaline treatment or tarry polymerization products in the escaping vapors from which the lubricating oils are condensed are avoided. The method of the invention is thus peculiarly effective, it is efficient and economical, it is satisfactory from the operating standpoint and it does not involve any unusual apparatus difficulties.

We claim:

1. In the distillation of petroleum stocks for the production of lubricating oils, the improvement which comprises vaporizing from the stock those constituents suitable as components of the desired lubricating oil, passing the vapors containing these constituents through a hot but exterally unheated liquid oil bath by introduction beneath the surface thereof, maintaining this oil bath alkaline by supplying caustic alkali thereto, discharging liquid from this bath, and taking off from this oil bath the components of the lubricating oil in the vapor state and condensing the same without permitting this condensate to contact as such with the liquid forming the oil bath.

2. In the distillation of petroleum stocks for the production of lubricating oils, the improvement which comprises vaporizing from the stock those constituents suitable as components of the desired lubricating oil, passing the vapors containing these constituents through a hot liquid oil bath, maintained at substantially constant volume, by introduction beneath the surface thereof, maintaining this oil bath alkaline by supplying caustic alkali thereto, discharging liquid from this bath, and taking off from this oil bath the components of the lubricating oil in the vapor state and condensing the same without permitting this condensate to contact as such with the liquid forming the oil bath.

3. In the distillation of petroleum stocks for the production of lubricating oils, the im-

provement which comprises vaporizing from the stock those constituents suitable as components of the desired lubricating oil, passing the vapors containing these constituents through a hot but externally unheated liquid oil bath by introduction beneath the surface thereof, maintaining this oil bath alkaline by supplying caustic alkali thereto, supplying steam to this oil bath, discharging liquid from this bath, and taking off from this oil bath the components of the lubricating oil and condensing the same without permitting this condensate to contact as such with the liquid forming the oil bath.

EUGENE C. HERTHEL.
HARRY L. PELZER.