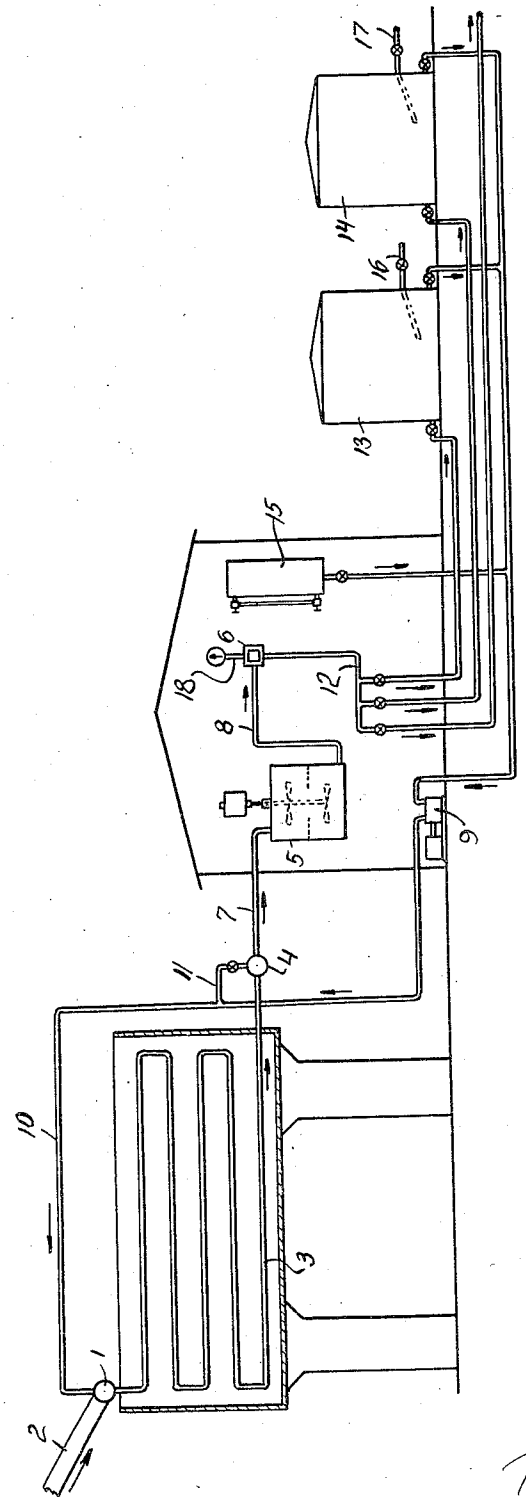
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ART OF REFINING

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ART OF REFINING

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1 Claim. (Cl. 196—32)

Our invention relates to improvements in the production of petroleum distillates, such as gasoline and kerosene, from sulphur-containing stocks, such as sulphur-containing crude petroleums.

Raw gasoline and kerosene distillates, for example, produced from sulphur-containing stocks frequently include sulphur compounds of a character such that the oxidation involved as a consequence of the usual access of air to the distillate converts these compounds either into free sulphur, or high boiling sulphur compounds, which forms of sulphur interfere with the usual sweetening operations and which are not easily removed without redistillation of the distillate or the use of unusual and expensive treating agents. This difficulty can be overcome, in some cases, by treatments applied, in effect, to the entire stock subjected to distillation, but as will be apparent such treatment of the entire stock subjected to distillation is not always expedient.

We have found that this difficulty can be overcome if the distillate, as such, is neutralized, substantially completely, prior to and while avoiding any oxidation of the distillate. We accomplish this, according to our present invention, by condensing a vapor mixture, including the distillate fraction, vaporized from the stock to form a condensate, substantially completely, prior to and while avoiding any oxidation of the condensate by positively dispersing an aqueous caustic alkali solution containing an excess of caustic alkali over that consumed in the neutralization in the condensate, and thereafter separating the condensate, constituting the distillate, from the aqueous solution. The dispersion of the aqueous caustic alkali solution in the condensate, to be effective, must be carried to an extent sufficient to effect the neutralization, and is advantageously effected by supplying the condensate and the aqueous solution to mechanical mixing means in proportions such that an excess of caustic alkali over that consumed in the neutralization is maintained in the mixing means and separating the condensate from the aqueous solution after discharge of the condensate mixture from the mixing means. We thus effect a special refining of the distillate, gasoline or kerosene for example, which eliminates or converts into a stable form those compounds which otherwise would tend to form free sulphur or high boiling sulphur compounds as a consequence of the oxidation to which we have referred and which, ultimately, is hardly to be avoided. This special refining, moreover, does not involve treatment of any part of the original stock other than the chosen distillate or distillates.

Neutralization of petroleum distillates including gasoline and kerosene with caustic alkali has been involved, as one step, in many refining operations hitherto proposed, but so far as we are aware such neutralization, as hitherto applied, has been ineffective to accomplish the purpose of our invention either because it was not carried to completion or because it was not applied prior to and while avoiding such oxidation of the distillate or because it was not completed before the distillate was exposed to such oxidation.

The invention will be further described in connection with the accompanying drawing which illustrates, diagrammatically and conventionally, in elevation and partly in section, one form of apparatus adapted for the practice of our invention.

Referring to the drawing, a vapor mixture including the distillate fraction from an appropriate still or fractionating system is delivered to the condenser header 1 through connection 2. This vapor mixture is subjected to condensation as it flows downwardly through condenser coils 3 connected between the headers 1 and 4. The condensate, and any uncondensed vapors and gases, flows from the header 4 through the mechanical mixer 5 to the look-box 6, through connections 7 and 8. The mechanical mixer comprises a chamber within which a pair of impeller-agitators mounted upon a motor-driven shaft and a baffling partition are arranged. The chamber itself is sealed and the connections 7 and 8 between the header 4 and the chamber and the chamber and the look-box 6, respectively, are arranged to maintain a liquid seal precluding access of air to the chamber. An aqueous caustic alkali solution is delivered, by means of pump 9, either to the header 1 through connection 10 or to the header 4 through connection 11. In either case the aqueous caustic alkali solution and the condensate flow together to the mechanical mixer 5 through connection 7. The concentration of the aqueous caustic alkali solution supplied to the mechanical mixer 5, and the rate at which it is supplied, are regulated, with advantage, so that an excess of caustic alkali over that consumed in the neutralization is maintained in the mechanical mixer 5. For example, this concentration and rate may be regulated so that the unconsumed caustic alkali in the condensate mixture discharged into the look-box 6 constitutes about 25–50% of the caustic alkali supplied to the header 1 or the

header 4. From the look-box 6 the condensate mixture is discharged through the manifold 12 to appropriate tankage in which the aqueous solution remaining is settled from the condensate constituting the distillate. The aqueous caustic alkali solution separated by settling, in the tanks 13 and 14 for example, is recirculated through the mechanical mixer 5 by means of pump 9, additional fresh caustic alkali being supplied to the recirculating aqueous caustic alkali solution, advantageously as a concentrated aqueous solution, from tank 15 to replace the caustic alkali consumed in the neutralization. The circulating aqueous solution is replaced in toto from time to time as it becomes fouled with reaction products. The separated condensate is discharged from the tanks 13 and 14 through swing-pipe connections 16 and 17, respectively. Connection 18 is provided for the collection of any uncondensed vapors and gases separated in the look-box 6.

That part of the apparatus described as a condenser in the preceding paragraph may instead function as a cooler, a condensate separated in an appropriate fractionating system instead of a vapor mixture being delivered to header 1 through connection 2. That is, an overhead vapor mixture from a fractionating tower may be delivered to the header 1 through connection 2 or a side stream, condensed in a fractionating tower, may be delivered to the header 1 through connection 2 in the apparatus illustrated. As will be apparent from the fact that our invention can be practiced in the apparatus illustrated by supplying the aqueous caustic alkali solution either to the header 1 or to the header 4, the caustic alkali solution can be introduced either into the condensate or into the vapor mixture from which the condensate is formed, the positive dispersion of the aqueous caustic alkali solution being effected with respect to the liquid condensate.

In applying our invention to straight run gasolines and kerosenes from Texas Panhandle crudes of high sulphur content, for example, we have supplied 15–20% by volume on the distillate of a 5° Bé. aqueous solution of sodium hydroxide with satisfactory results. The amount of caustic alkali, as such, necessary to maintain an excess over that consumed in a neutralization is easily determined, the amount of any aqueous solution of caustic alkali necessary to supply this amount of caustic alkali then being determined by the concentration of the particular solution. In general, it is advantageous to use a relatively large volume of a dilute solution. When recirculating the aqueous solution including unconsumed caustic alkali separated from the distillate by settling, the make-up caustic alkali may be supplied, for example, as a 40°–45° Bé. aqueous solution of sodium hydroxide. In the particular operations to which we have just referred, about 50–75% of the caustic alkali in the aqueous solution supplied was consumed per cycle. The gasoline distillate so treated was finished by conventional sweetening and the kerosene distillate so treated was finished by treatment with 10 pounds per barrel of 66° Bé. sulphuric acid followed by conventional sweetening.

We claim:

In the production of straight run petroleum distillates by distillation from sulphur-containing stocks, the improvement which comprises condensing a vapor mixture, including the distillate fraction, vaporized from the stock to form a condensate and thereafter neutralizing the condensate, substantially completely, prior to and while avoiding any oxidation of the condensate by positively dispersing in the condensate a dilute aqueous solution of sodium hydroxide containing an excess of sodium hydroxide over that consumed in the neutralization, and thereafter separating the condensate, constituting the distillate, from the aqueous sodium hydroxide solution.

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