

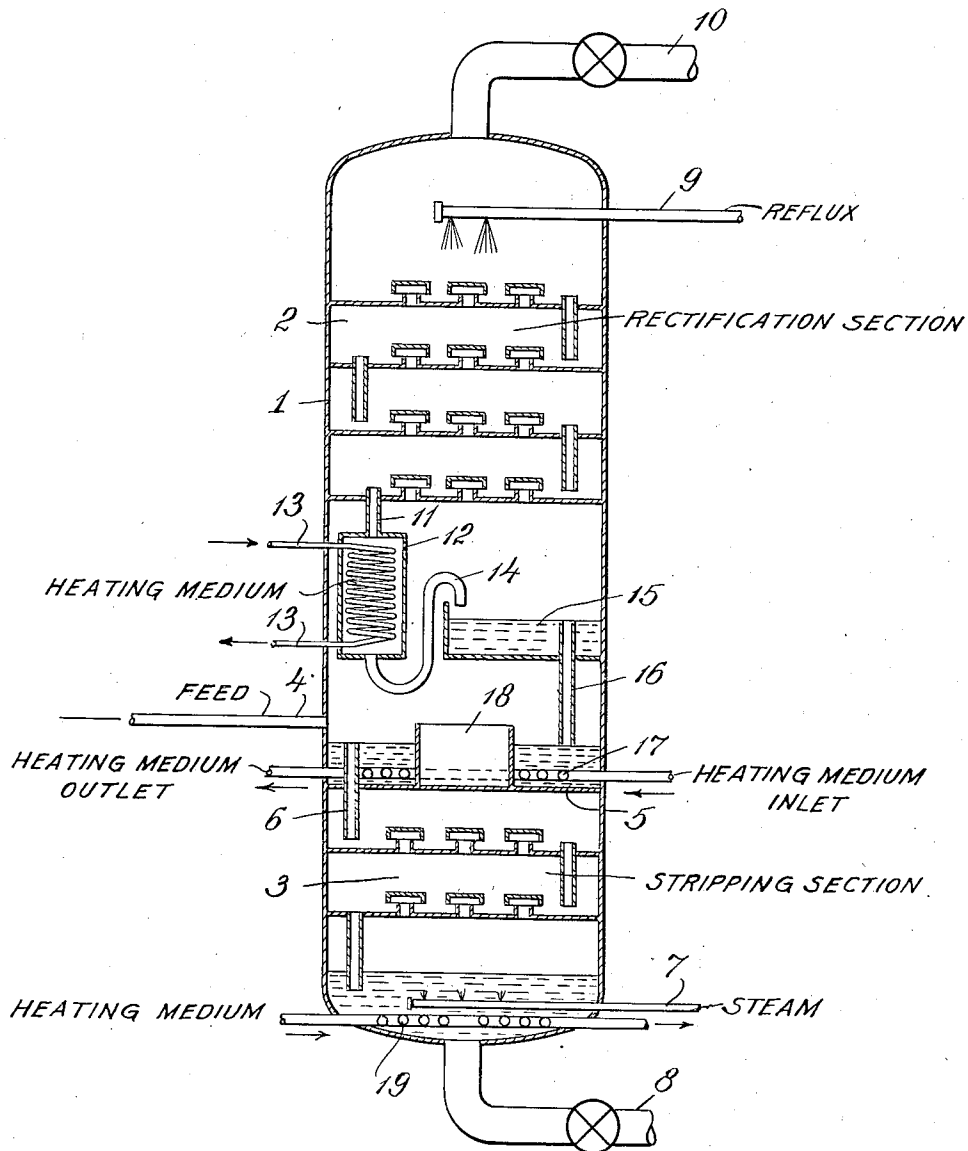
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METHOD OF DISTILLATION

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METHOD OF DISTILLATION

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1 Claim. (Cl. 202—40)

In United States Patent No. 2,113,130, issued to me on April 5, 1938, assigned to Socony-Vacuum Oil Company, Incorporated, I have disclosed a method of distillation capable of securing independent control over the amount of liquid descending through the rectifying plates of a fractionating tower which are located above the point of feed, and the amount of liquid descending through the stripping plates of that tower which are located below the point of feed.

This invention has to do with improvements in method over the invention disclosed in said United States Patent No. 2,113,130.

As pointed out in that patent, the most complete rectification is attained when the ratio of liquid passing down over the rectification plates is relatively great in proportion to the vapor going up, while in the stripping plates the best stripping is attained when the ratio of liquid to vapors is relatively low. In the ordinary tower conditions at the feed plate result in adding to each section of the composite tower a portion of ingredient which serves to alter the desired liquid to vapor relation in a direction away from most efficient operation. My prior patent discloses broadly a method of avoiding this by collecting the downflowing liquid within the tower at a point between rectification and stripping, adding heat to it, and thus vaporizing and preventing entry to the stripping section of those portions of the liquid which normally belong to the liquid refluxing upon the rectification trays.

While the general method offers a great improvement over the prior art, I have found that in certain applications the degree of control afforded by the method set forth therein was not sufficiently complete, and have devised the presently disclosed method and apparatus to overcome those difficulties.

This invention has for an object the provision of a method of distillation capable of performing separation between materials of different boiling point of a high degree of accuracy.

A further object is the provision of additional steps of reheating and vaporization within the usual composite tower housing both rectification and stripping operations.

As an example of operation wherein a precision of operation is desirable which has not heretofore been obtainable or obtainable only with difficulty, I may cite the preparation of heavy charging stock for cracking from crude. In such operation, it is desirable to separate as much overhead stock for cracking charge as possible, but it must be entirely free of materials

of asphaltene or asphalt-like nature, since even a very small amount of these, if present in the cracking stock, gives rise to deposits of coke at the point of cracking which are disproportionately large.

In my prior patent the liquid portion of the feed is heated together with the liquid descending the tower. While sound in principle, this does not permit of the precise operation now desired, or if it be so operated to secure that precision, requires the use of large amounts of heat, since to free the liquid portion of the feed entirely from the material which should be sent back up the tower, I am obliged to vaporize a considerable portion of the material which should remain liquid and be sent direct to the stripping section.

Accordingly, I have now provided a process wherein the liquid descending from the rectification plates is separately collected, heated and revaporized, so that no substantial portion of it comes in contact with the liquid portion of the feed, and have additionally provided a step of heating the liquid at just below the point of feed, to assist in complete separation of the feed and reflux constituents and their progress into the proper portion of the tower.

In order to understand my invention, reference is made to the drawing attached hereto, wherein there is shown in diagrammatic form, an apparatus appropriate for the practice of my invention.

In the single figure of this drawing, 1 denotes a composite fractionating tower, consisting of a rectification section 2 and a stripping section 3. Heated feed is supplied to the tower by pipe 4 and within the tower divides into liquid and vapor, the vapor portion of the feed rising through rectification section 1, while the liquid portion of the feed collects within trap tray 5 and subsequently passes through overflow 6 and down through stripping section 3, where it is stripped by the agency of steam or other stripping medium introduced through pipe 7, the final stripped bottoms product leaving the tower through pipe 8.

The vapors passing upwardly through rectification section 2 are subjected to fractionation by contact with a "wet" or liquid reflux produced by introducing a proper material, such as condensed overhead product, etc., through pipe 9. The fractionated overhead product leaves the tower through pipe 10. Complete fractionation, such as, for example, that necessary to remove the last traces of coke-forming constituents from

overhead stocks to be used as charge for vapor phase cracking systems, requires the introduction of a relatively copious amount of reflux liquid at 9 and consequently a relatively large amount of liquid flowing down through the trays in rectification section 2. For best operation of stripping section 3, none of this liquid from section 1, except the heavy material which has been fractionated from the vapors, should pass into section 3. Reflux liquid from section 2 should be vaporized at or near the point of feed, but if, when the high rates of reflux are used, it is attempted to vaporize this reflux liquid from a mixture of reflux liquid and feed, it is difficult to secure complete vaporization of reflux liquid without overvaporization of feed. To correct this condition, I have now provided two separate heating and vaporization steps within the tower at the point of feed, which operate as follows: Downflowing reflux liquid from the bottom tray of rectification section 2, passed through pipe 11 into heater 12, wherein it is heated to a vaporizing temperature by a heating material handled through pipes 13. Reflux liquid then heated passes through pipe 14 into open tray 15, wherein a substantial portion of the reflux liquid vaporizes, leaving in tray 15 only a small portion of unvaporized reflux liquid together with the unvaporized heavy oils which have been removed from the vapors within the rectification section. This oil from tray 15 then passes through overflow pipe 16 to tray 5, where it joins the liquid portion of the feed. To secure revaporization of such reflux as remains liquid, and to secure separation of liquid portions of the feed from portions which should be vaporous at this point, but may not have separated for known reasons, such as high latent heat demand, tray 5, is provided with a heating coil 17 through which a heating medium is passed. Vapors from the stripping section 3 may pass upward through tray 5 without contacting the oil therein by some means such as chimney 18.

The liquid collecting at the bottom of tower 1 is preferably additionally heated by heating coil 19.

The device and process thus shown is applicable to many uses in the distillation of oils. For example, suppose it were desired to separate a residual crude stock into a heavy charging stock for vapor phase cracking and at the same time recover from the bottoms a heavy asphalt highly free from cracked products resulting from the exposure of the asphalt for long times to high temperatures. The feed stock may be heated, during a short time, to a temperature high enough to secure proper separation, that separation may be conducted and completed in tray 5, and the liquid from tray 5 passing through section 3 can be steam stripped at comparatively low temperatures and times without the extensive steaming necessary were not liquids from section 2, (other than those desired) entirely prevented from entering section 3. At the same time, section 2 may be operated at reflux rates sufficient to entirely fractionate from the charging stock being prepared every trace of coke-forming bodies.

A further important advantage is apparent in the operation of this invention in that the flow of residue through the apparatus is conducted

at a maximum rate at all times. For example, the distillation residue in tray 5 is held to a minimum quantity and consequently the flow through this tray 5 is rapid and the exposure of oil therein to temperatures capable of bringing about decomposition is at a minimum. If material from the upper section of the tower, descending through pipe 11, were added to the liquid in tray 5 without intervening substantial vaporization, this result could not be obtained. This feature is of particular importance, as will be appreciated, in the manufacture of lubricating oils and asphalts.

Furthermore, this method of operation and this form of apparatus, by permitting contact of tower liquids with heating media for minimum, controlled times, permits the use of high temperature heating media not heretofore thought possible to use in fractionating, etc., and so extend the usefulness of this method into the field of heavy oil. In particular, the manner of heating and of exposing only small amounts of oil to such heating permits the use of heating media in coils 19, 17, and heater 13, which has temperatures of the order of 800° F. or even higher. This will permit of the use of hot oil streams from cracking operations. In some recently developed cracking methods, use is made of fused inorganic salts as heat exchange media and this method and apparatus is particularly adapted for the use of such media.

I am of course aware that other forms of apparatus may be used to embody my process, such as, for example, the use of external "reboiler" bodies to perform the function of tray 5 and heater 12. All of these, in so far as novel, I claim as my invention, subject to the limitations expressed in the following claim.

I claim:

A process of fractional distillation comprising the steps of heating a feed stock to a temperature at which it is at least partially vaporized, introducing the heated feed into a fractionating tower intermediate the ends thereof and separating it therein into vapor and liquid fractions, subjecting the vapors to rectification above the point of feed by contact with reflux liquid, collecting the downflowing reflux liquid at the base of the rectification section above the point of feed, heating said collected reflux liquid to a temperature sufficient to cause substantial vaporization thereof and effecting such vaporization, all prior to any contact of reflux liquid and feed liquid, collecting the remaining reflux liquid and the liquid portion of the feed in a pool immediately below the point of feed, supplying heat to the liquid in said pool, passing a portion of the liquid from the pool to the section of the fractionating tower below the point of feed, contacting it therein with a stream of rising vapors, passing said stream of rising vapors into the bottom of the rectifying portion of the tower, collecting remaining liquid in a pool at the bottom of said tower and supplying heat to said pool to generate such rising stream of vapors, whereby the rectification and stripping sections may be operated independently and whereby reflux liquid from rectification is substantially revaporized without contact with liquid portions of the feed.

GEORGE S. DUNHAM.