Aug. 26, 1941. W. W. KRAFT 2,254,182

DISTILLATION PROCESS

Filed Aug. 12, 1939

Fig. 1.

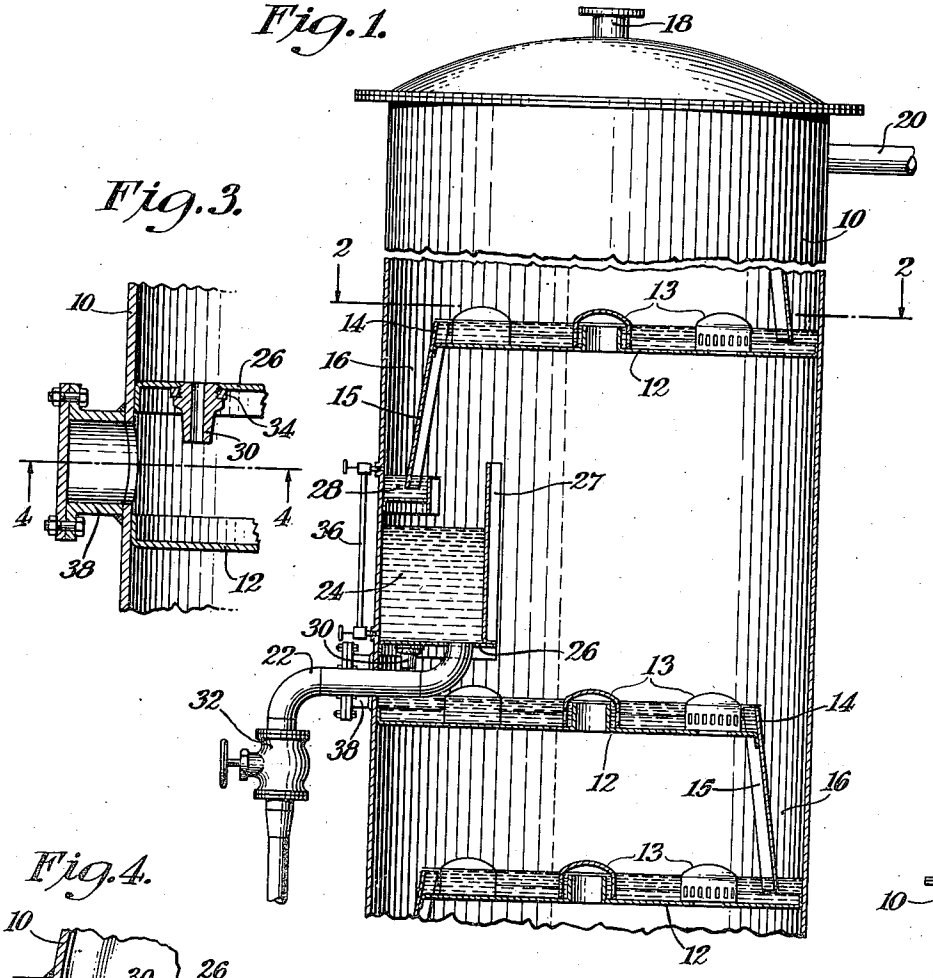


Fig. 3.

Fig. 4.

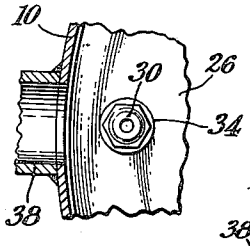
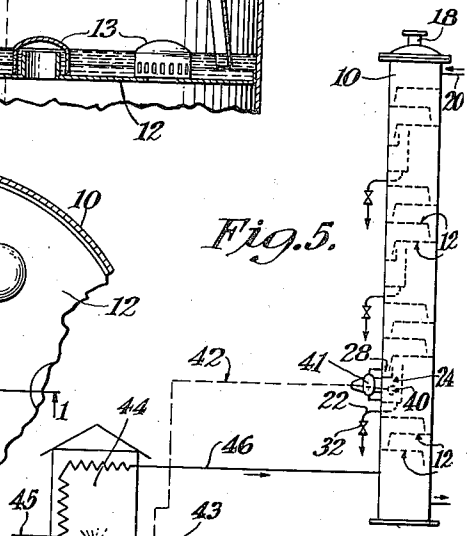


Fig. 2.

Fig. 5.



INVENTOR
Wheaton W. Kraft
BY Nathaniel Ely
ATTORNEY

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DISTILLATION PROCESS

Wheaton W. Kraft, New York, N. Y., assignor to The Lummus Company, New York, N. Y., a corporation of Delaware

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5 Claims. (Cl. 202—40)

This invention relates to an improved method of and apparatus for controlling the composition and the quantity of side streams withdrawn from a distillation column or the like, and to the method of controlling the fractionation in said column.

In the distillation of a liquid mixture composed of a number of constituents having different boiling points or different boiling ranges, it is frequently customary to withdraw from the column a certain portion or fraction of this mixture as a side stream or as multiple side streams. Since the composition and quantity of the side streams must generally meet certain required specifications, careful control of the operating conditions within the column is essential. Control of side stream composition is a function of the size of the stream and the degree of fractionation practiced, which latter is dependent upon the number of fractionating decks and the heat content of the column charge. This heat content in turn determines the amount of reflux made available to the column. Control of the quantity of the side stream depends on the means employed to divide the liquid downflow at the point of side stream withdrawal from the column.

The use of a device to proportionally divide the liquid downflow in a column for the removal of a side stream therefrom has heretofore been suggested. It will be apparent, however, that with a variation in the amount of reflux to the column, the ratio of side stream to charge will accordingly change, whereas the proportion of side stream to reflux will remain constant. Since a side stream normally represents a certain desired fraction of the charge material, it is essential to maintain the ratio of side stream to charge constant. With a proportional division of liquid downflow, additional means to control the quantity of the side stream withdrawn must also be provided.

The principal object of the invention is to provide an improved method of controlling the fractionation in a distillation column and the removal of side streams of predetermined quantity therefrom, whereby variations of the operating conditions in the column do not affect the ratio of the side stream to the column charge.

A further object of the invention is to provide an improved apparatus to facilitate the removal of a side stream from a distillation column, such apparatus comprising means for the continuous removal of the side stream at a constant rate and means so calibrated that control of the amount of reflux at the point of side stream withdrawal may be obtained by regulating the heat input to the column, said apparatus being relatively inexpensive, easy to maintain, accurate within the limitations required, and adjustable to suit desired conditions.

A still further object of the invention is to provide a control for the rate of firing of a still whereby the heat input is varied in accordance with the volume of liquid downflow at any desired point of the distillation column, such as the first side stream above the feed, whereby accurate control of fractionation may be obtained and dry decks avoided.

Further objects and advantages will appear from the following disclosure of a preferred form of embodiment of my invention, taken in conjunction with the drawing illustrative thereof, in which,

Fig. 1 is an elevation with parts broken away and parts in section, of a portion of a distillation column, the section being substantially on line 1—1 of Fig. 2.

Fig. 2 is a partial horizontal cross section, taken substantially on line 2—2 of Fig. 1.

Fig. 3 is an enlarged detail, in vertical section, of a part of the column showing the arrangement of the reflux outlet from the liquid downflow collecting zone.

Fig. 4 is a horizontal section taken substantially on line 4—4 of Fig. 3, and

Fig. 5 is a diagrammatic view of a still and distilling column showing an automatic control.

According to my invention, a collecting zone is provided in a distillation column for the collection of the entire liquid downflow at the point of withdrawal of a side stream therefrom. This zone is provided with two outlets, one for the removal of a portion of the liquid downflow as the side stream, and the other for the continuation of the remainder of the liquid downflow to the next lower fractionating zone of the column. The side stream outlet is equipped with suitable means so that a constant amount of liquid is continuously withdrawn therethrough, irrespective of variations in operating conditions which affect the amount of liquid flowing into the collecting zone. Control means are also provided to vary the rate of heat input to the column to always maintain a depth of liquid in the collecting zone, whereby the degree of fractionation of the side stream is subject to a considerable degree of control. It will be understood that the depth of liquid is a direct measure of the reflux flow to the next lower fractionating zone, and with the

arrangement used, the head of liquid over the reflux opening can be determined.

The drawing illustrates a typical distillation column, generally indicated at 10, which is provided with a plurality of fractionating decks 12, having suitable bubble caps 13, such decks being spaced at any desired interval appropriate to the character of the material undergoing fractionation. As is customary, each deck is provided with a weir 14, so that liquid may collect thereon to the desired depth, and a down flow passage 16, so that liquid may flow downwardly through the column from deck to deck. Weir 14 may be conveniently extended downward, so that its lower portion 15 serves to define downflow passage 16. Means for the removal of the overhead vapors for condensation thereof are provided at 18, and means for returning a portion of the vapor condensate as reflux are provided at 20. A drawoff 22 is provided for the withdrawal of a side stream from any desired section of the column.

In accordance with a preferred form of embodiment of my invention, all the liquid downflow from the deck immediately above the point at which the side stream is to be withdrawn is collected in a receiver 24, conveniently formed by the plate members 26 and 27, which are suitably fastened to the internal walls of the column.

The liquid downflow is first collected in a trap 28, from which it overflows into receiver 24, in order to prevent vapors from rising through liquid passage 16. As an additional means of preventing the flow of vapors through passage 16, trap 28 may be so located that its top edge is somewhat lower than the top edge of plate 27. In order to properly accommodate the receiver, the distance between the decks between which receiver 24 is inserted is preferably made somewhat greater than that between the other decks.

Receiver 24 is preferably provided with two outlets, one of which is a connection to the side stream drawoff 22, and the other of which is a short tube 30 for carrying the balance of liquid downflow as reflux to the next lower fractionating zone of the column. A valve 32 is conveniently installed in drawoff 22 to control the flow of liquid therethrough, and is made easily adjustable so that the amount of liquid withdrawn as the side stream may be varied as desired. As is well understood, this valve is customarily placed in a location convenient for hand control, or even may be automatically controlled.

The tube 30 which may be conveniently screw threaded into boss 34, which is attached to plate 26, is preferably of such length that it acts as an orifice, and may be calibrated before being secured to receiver 24, so that the head on the tube 30 will indicate the quantity of reflux which is furnished to those fractionating decks below the side stream drawoff. Variations in the height of liquid in receiver 24, however, will have a negligible effect on the volume of side stream withdrawn, inasmuch as the liquid height maintained in receiver 24 is relatively small as compared to the vertical distance between the side stream drawoff and the side stream receiver. Such variations, however, will affect the amount of liquid refluxed to the column section below the drawoff deck. A gauge glass 36 may be interconnected into column 10 above the side stream drawoff 22, so that the height of liquid in receiver 24 may be directly determined.

The operation of my invention is as follows: A complex liquid mixture, heated sufficiently so that part of it is in the vapor state, is introduced into column 10 at a point below the side stream drawoff 22. Overhead vapors are taken off at 18 and condensed, a predetermined portion of the condensate being removed as product and the remainder being returned to the column at 20 as reflux. A part of the downflow liquid is withdrawn as a side stream through drawoff 22, positioned sufficiently below the top of the column so that suitable fractionation is obtained in the upper section of the column, and the remaining portion is allowed to flow through orifice 30.

The side stream withdrawn through line 22 represents the percentage of the liquid charged to the column that is desired as a product. With valve 32 set accordingly, only this amount of liquid will continuously flow through line 22, regardless of the quantity of reflux flowing into receiver 24.

Since the amount of the side stream is fixed in accordance with the product specifications as well as by the known yield of this material based on charging stock, close control of fractionation is desirable. The degree of fractionation obtained in the section of the column above drawoff 22 determines the initial boiling point of the side stream, whereas that obtained in that part of the column below drawoff 22 determines the end boiling point of the side stream. By adjusting the liquid level in receiver 24, the boiling range of the material withdrawn through line 22 can be controlled. The flow through orifice 30 determines the degree of fractionation obtained, by giving an indication of the amount of reflux supplied to the column below drawoff 22. Regulation of the heat input of the column charge, whereby the quantity of vapors and of reflux vary accordingly, enables a suitable control of fractionation to be effected.

Although the calibration of the orifice tube 30 will not be of the order of magnitude obtained in precision metering instruments, it will be sufficiently accurate to provide an entirely satisfactory control of the fractionation in column 10. It furnishes a readily available means of determining the degree of fractionation between the side stream and the next heavier stream, whether another side stream or the column residue. Preferably, the indication of liquid level is made at the first side stream above the feed, for if there is available reflux liquid there, ordinarily there is reflux at all higher decks. It may be used at every drawoff, however.

It will be appreciated that gauge glass 36 is not the only means by which the liquid level in receiver 24 may be determined. As shown in Fig. 5, a float 40 may be positioned in the receiver and so equipped with a controller 41 that the liquid level therein will be indicated at a considerable distance from the column, as for example in the control room whereby the heat to the charge stock may be varied accordingly. If desirable, for a certain liquid under definite operating conditions, the liquid level controller 41 may be so connected as by line 42 to a fuel supply 43 in heater 44, that the temperature of the charge stock entering at 45 and discharging through line 46 will be regulated automatically. It will be understood that the drawing illustrates in detail only the means for side stream withdrawal and downflow measurement. The actual baffle arrangement on the deck immediately below the reflux orifice 30, necessary to avoid turbulence and to break up any

foaming which might be encountered, is not shown. It is also to be understood that reflux orifices of varying diameter may be employed with changes in the character of the liquid undergoing fractionation. Hand-hole 38 is provided for the removal and insertion of such orifices.

My invention presents a simple method of effecting the continuous withdrawal of a side stream representing a constant percentage of column charge combined with control of operating conditions in the column to obtain the desired degree of fractionation. Furthermore, by means of the indication of the liquid level in the reflux receiver 24, close control of operating conditions necessary to obtain the desired degree of fractionation is possible.

While I have shown a preferred form of embodiment of my invention, I am aware that modifications may be made thereto and I, therefore, desire a broad interpretation of my invention within the scope and spirit of the claims appended hereinafter.

What I claim is:

1. In a fractionation apparatus having a plurality of fractionating decks therein, means to feed a stream to a lower part of said apparatus, and means to form reflux for said respective decks, the combination of means to collect all of the downflowing reflux between certain adjacent decks said means being positioned above the point of feed, means to remove from said collecting means a fixed percentage of the feed as a side stream, said collecting means having an orifice communicating with the next lower deck, the flow through such orifice being proportional to the head in the collecting means, and means to measure the head of liquid in said collecting means to indicate the rate of flow of the balance of the collected reflux to the fractionating deck below the point of side stream drawoff.

2. In combination with a distillation column having a plurality of fractionating plates therein, a heater to heat the charge therefor, means to introduce the heated charge into a lower part of said column, a receiver positioned between certain adjacent plates within said column above the charging point to receive liquid downflow from the plate above the receiver, means to withdraw from said receiver a portion of said liquid as a side stream as a constant percentage of the charge to the column, said receiver having a calibrated orifice communicating with the plate below said receiver, the flow through such orifice being proportional to the head in said receiver, means to measure the head of liquid in said receiver to indicate the rate of flow of the balance of the liquid to the plate below said receiver, and means to control the heat input to the charge according to variations in the rate of liquid flow through said orifice.

3. In combination with a rectifying column of the class described having a plurality of fractionating plates therein, means to feed a heated liquid to a lower part of said column, means to reflux the column, means to collect the downflowing reflux liquid from an intermediate fractionating plate above the point of feed, means to remove a portion of said reflux liquid from said collecting means as a side stream as a constant percentage of the feed to the column, said collecting means having a calibrated orifice communicating with the lower portion of the column, the flow of reflux liquid through said orifice being proportional to the actual height of liquid in said collecting means, the actual height of liquid in the collecting means above the side stream withdrawal means and the orifice being the same, said reflux liquid stream passing through the orifice having a low effective head variable with said actual height of liquid and said side stream having a relatively high effective head maintained relatively constant irrespective of said actual height of liquid, and means to measure said actual height of liquid to determine the rate of flow of reflux liquid to the plate below the point of side stream drawoff.

4. The method of fractionation, which comprises heating a liquid, feeding said heated liquid to a lower part of a distillation zone containing a plurality of fractionating decks, said distillation zone containing downflowing reflux liquid and rising vapor in direct countercurrent contact at said decks, collecting the reflux liquid passing from an upper deck to a lower deck in an intermediate collecting zone above the point of feed, removing a definite, constant proportion of the feed as a side stream from said collecting zone, passing the remainder of the reflux liquid to the lower deck through an opening in said collecting zone communicating with said lower deck, the flow through such opening being proportional to the head of liquid in said collecting zone, measuring the head of liquid in said collecting zone to indicate the rate of flow of reflux liquid to the lower deck, and controlling the heat input to the feed in accordance with variations in the rate of flow of reflux to the lower deck.

5. In combination with a fractionating column having fractionating decks therein and having means to feed a stream of liquid to a lower part of said column to be fractionated and in which the fractionation is accomplished by countercurrent contact of reflux liquid and vapors, the improvement which comprises means to collect all the reflux liquid passing downwardly from one intermediate deck to another above the point of feed, said collecting means having an opening communicating with the next lower deck, the flow through said opening being proportioned to the head of reflux in said collecting means, means to measure the head of reflux in said collecting means to indicate the rate of flow of reflux to the lower deck, and means to remove a fixed percentage of the feed as a side stream from said reflux collecting means.

WHEATON W. KRAFT.