Jan. 4, 1938. P. J. ROELFSEMA 2,104,310

METHOD OF FRACTIONATION

Filed July 14, 1934 3 Sheets-Sheet 1

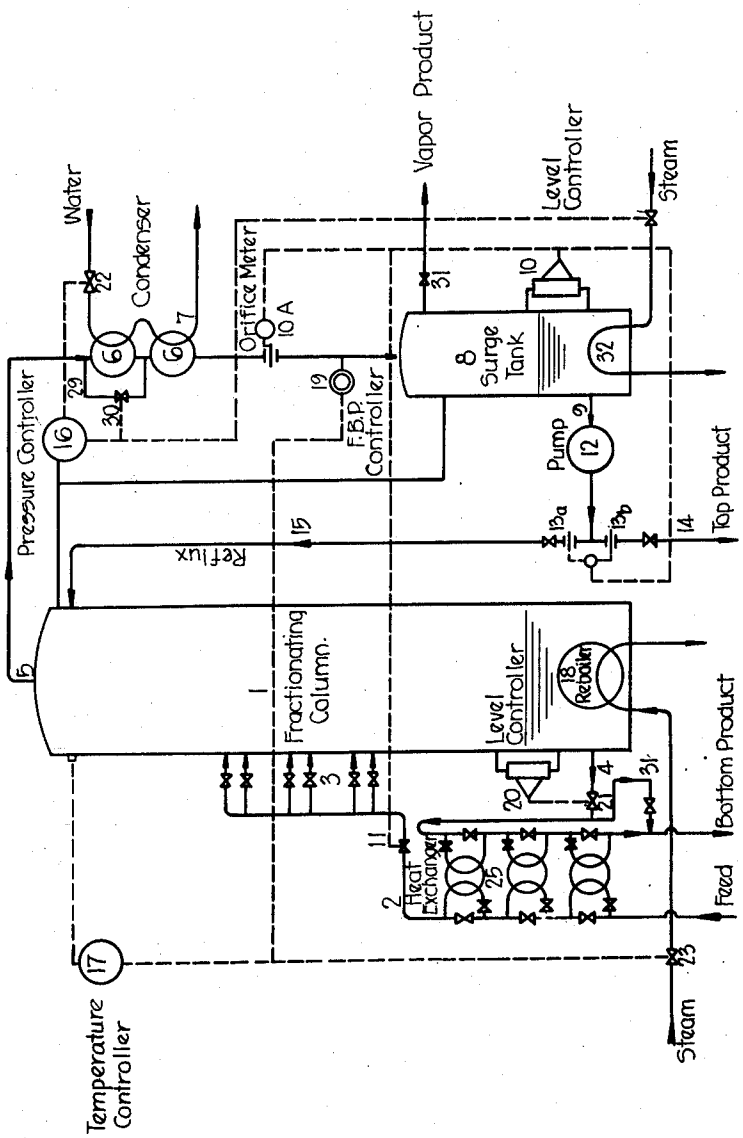


FIG. 1

Inventor : Petrus J. Roelfsema

By his Attorney

Jan. 4, 1938. P. J. ROELFSEMA 2,104,310

METHOD OF FRACTIONATION

Filed July 14, 1934 3 Sheets-Sheet 2

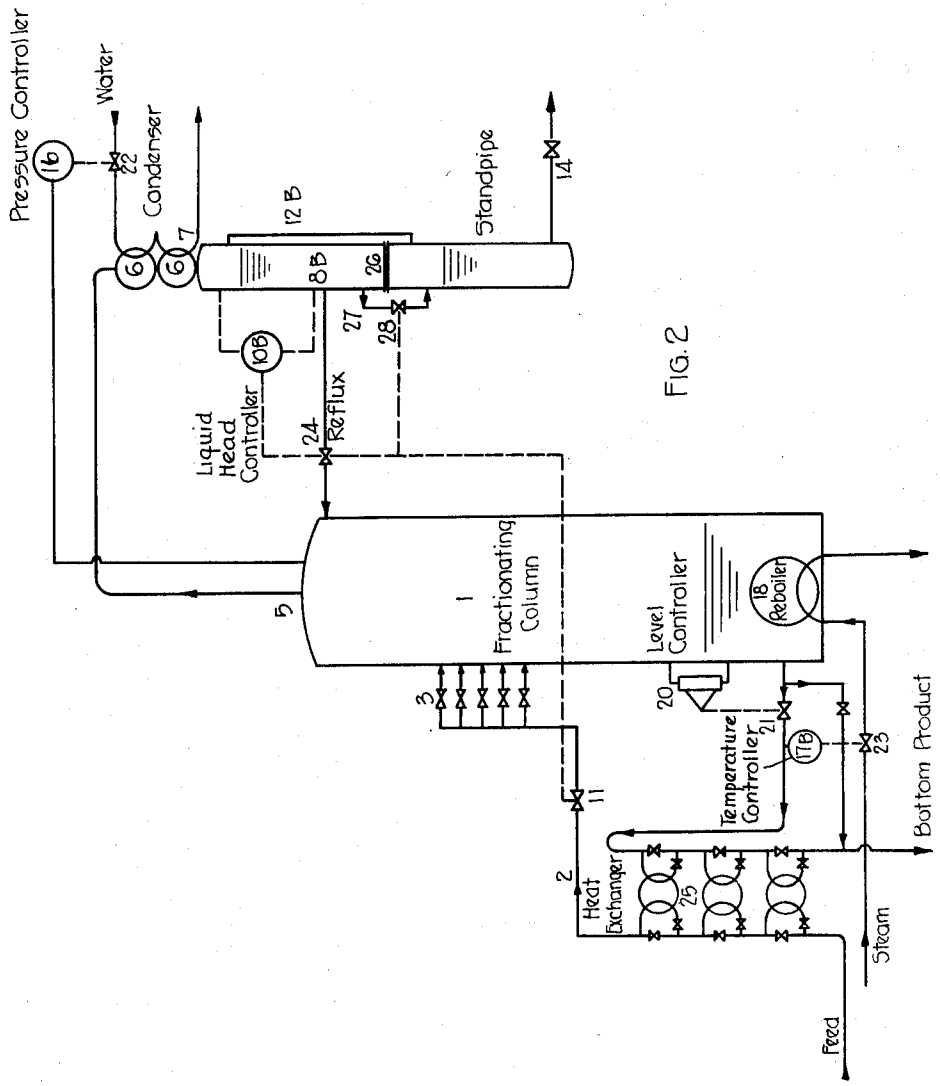


FIG. 2

Inventor : Petrus J. Roelfsema

By his Attorney B. Gratama

Jan. 4, 1938. P. J. ROELFSEMA 2,104,310

METHOD OF FRACTIONATION

Filed July 14, 1934 3 Sheets-Sheet 3

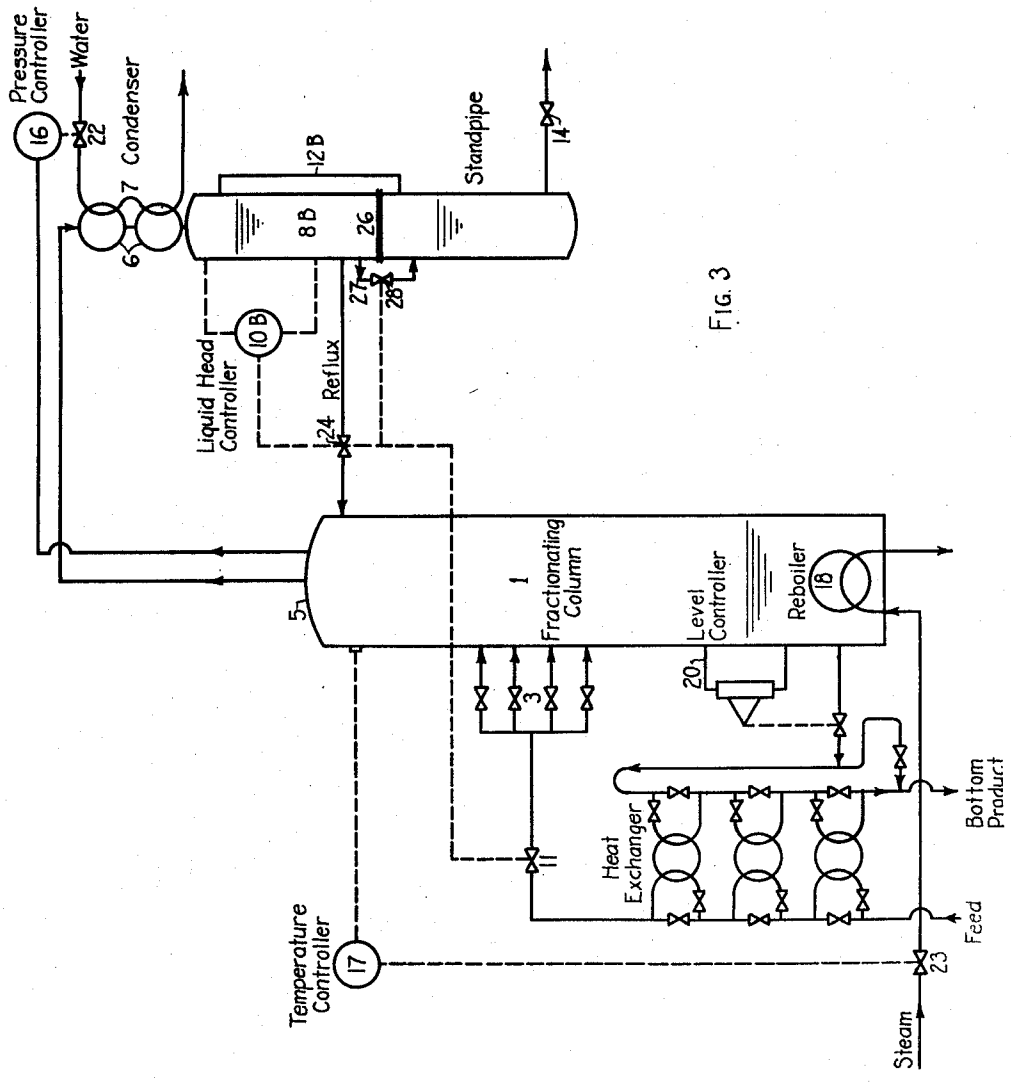


FIG. 3

Inventor: Petrus J. Roelfsema

By his Attorney:

Patented Jan. 4, 1938 2,104,310

UNITED STATES PATENT OFFICE

2,104,310

METHOD OF FRACTIONATION

Petrus Jurjen Roelfsema, Walnut Creek, Calif., assignor to Shell Development Company, San Francisco, Calif., a corporation of Delaware

Application July 14, 1934, Serial No. 735,216

2 Claims. (Cl. 196—72)

This invention pertains to methods of fractionation and is more particularly concerned with means of reducing, separately or simultaneously, the fluctuations in the properties of the product, the load on the fractionating apparatus, and/or increasing the yield of desired products.

In the ordinary practice of fractionating petroleum distillates, the composition of the feed unavoidably fluctuates within certain often very wide limits. These fluctuations in the feed composition may cause corresponding changes in the load on the apparatus and in the properties and yields of the product. Under such conditions, a fractionating column will operate at a load which will continuously vary, operations being carried out at a load higher or lower than the optimum desired.

If the percentage of volatile components present in the feed decreases and the reflux remains constant, the load on the column will decrease to one lower than the optimum. At the same time the reflux ratio will exceed the one corresponding to a desired and economical yield, so that the process becomes wasteful of heat.

If the percentage of volatile components in the feed increases and the reflux remains constant the load on the column will exceed the optimum and at the same time the reflux ratio will be lowered and the process will then fail to produce the desired economical yield.

Numerous methods of control and regulation have been devised to preclude the occurrence of such undesirable conditions in the operation of a fractionating system, these methods being directed to correlating the conditions of temperature, pressure, heat input, etc., with the desired compositions and rate of flow of the material passing to, through and from the system.

It is, however, believed that the prior art has failed to make a proper distinction between the following three main variables:

(1) Properties of product, such as the final boiling point, the dew point, the initial boiling point, etc.

(2) Yield of product as determined by the reflux ratio,

(3) Load on the column as determined by the amounts of reflux and top product.

These variables were usually regarded as essentially interdependent, and a good control of one of them was achieved only at the sacrifice of another.

The three-fold object of this invention is, therefore, to control the specified properties of the final product, the amounts of yield, and the load on the apparatus, as factors largely, if not entirely, independent from each other.

First, the specified properties of the desired product are maintained constant by regulating the heat input for a fixed working pressure as a function of the controlled significant temperature of the system.

Second, the yield of the product as expressed in % of the feed is maintained constant and preferably at its optimum value by maintaining a constant ratio between the amount of reflux returned to the column and the amount of top product withdrawn from the system. The control of the yield is assured in this way as long as changes of composition in the feed do not cause surges too large for the column to handle.

Third, the load on the apparatus is maintained constant and preferably at its optimum value by regulating the feed as a function of the total amount of overhead, i. e. reflux plus top product. Since the load on the column is determined by the sum of reflux and top products in mols, provided, as is usually the case, that the down-spout capacity is ample, the control of this sum will control the load. No direct control of the sum of mols of reflux plus top product being always practicable, it was discovered that an almost ideal approach is made by controlling the sum of the gallons of reflux plus gallons of top product.

My invention will be fully understood from the following description taken with reference to the drawings of Figs. 1, 2, and 3, which represent diagrammatically and partly in cross-section, three preferred forms of apparatus for practicing the process.

Referring to Fig. 1, the apparatus consists of a fractionating column 1, provided with suitable rectifying means, such as plates or packing, not shown; the column is also provided with a pipe connection 2 and a manifold 3 for introducing the feed, after passing it, if desired, through the heat exchangers 25, or any other form of heating apparatus. Steam or any other suitable heating agent may be delivered to the fractionating column in any conventional way, such, for example, as by means of a reboiler 18 as shown on the drawings. The rate of flow of the heating medium is regulated as a function of the temperature of the vapor near the top of the column by means of controller 17, or as a function of the final boiling point of the condensed vapor phase by means of controller 19, either of which may be adapted to regulate valve 23. Liquid

and vapor withdrawal pipes 4 and 5 are provided respectively near the bottom and top of the column. The withdrawal of the bottom product through pipe 4 is regulated by a valve 21, responsive to the liquid level controller 20. The vapor pipe 5 passes through a partial or total condenser 6, having means 7 for circulating a cooling medium automatically controlled by a valve 22, actuated by controller 16, which is responsive to the pressure in column 1 or tank 8. As an alternative, the same effect may be obtained by means of a by-pass pipe 29 controlled by a valve 30 or by a heating coil 32 in the surge tank 8. The condenser is connected with a surge tank 8, which is provided near its top with a vapor pipe 31, through which, if desired, the final product may be withdrawn in vapor form, in case that only a partial condensation is effected. Tank 8 is also provided with a liquid level controlling means 10. This means 10 is adapted automatically to actuate the valve 11 on the feed line 2 in such a manner as to restrict the feed flow whenever the liquid level in the tank 8 rises, or correspondingly to increase said flow when the liquid level in tank 8 falls.

As an alternative, instead of controlling the feed as a function of the rate of flow of condensate to the surge tank 8 indirectly, that is, through the intermediary of the liquid level controller 10, the rate of feed may be controlled directly as a function of the rate of flow of the condensate by installing an orifice meter 10A between the condenser and surge tank 8 and connecting it in a way identical to that of controller 10. This liquid flow meter will in this case perform all the functions of the liquid level controller 10, which may accordingly be dispensed with.

A pump 12 is provided to withdraw the condensed distillate at a constant rate from the tank 8 through pipe 9. A reflux pipe 15 connects the pump 12 with the top of the column 1 and a pipe 14 is used to withdraw the top product from the system in case that total condensation is effected. Flow ratio control valves 13*a* and 13*b* regulate the flow in pipes 15 and 14 respectively.

In cases where column 1 has sufficient capacity to handle considerable surges, and feed regulation by means of controller 10 and valve 11 is not necessary, valves 13*a* and 13*b* may be made responsive to controller 10 to maintain a constant ratio between the top product and the reflux and varying their sum in accordance with the rise or the fall of the liquid level in tank 8, i. e. in accordance with the rate of flow of the condensed top product.

The following separate automatic control and regulation means may be provided.

For regulating the desired properties of the top product, a temperature control means 17 or a final boiling temperature controller 18, such, for example, as disclosed in J. F. M. Taylor's Patent 1,876,364, may be used to regulate the flow of heating medium through a valve 23 into the reboiler 18. If a fully automatic control and regulation such as described is not desired, the same results may be approached by recording either the dewpoint temperature or the final boiling point and by manually regulating the flow of the heat medium to the reboiler in accordance with the recorded temperatures of the overhead or the top product.

Fig. 2 represents another embodiment of my invention particularly suited when the composition of the bottom product is to be controlled. In this case, a temperature control means 17B may be used to maintain a predetermined temperature at the bottom of the column in accordance with the working pressure and with the desired physical properties of the bottom product. While this assures a continuous production of a specification bottom product, the simultaneous automatic control of the rate of feed by a master controller or liquid head controller, 10B maintains the operation of the column at a constant load.

In the operation of the illustrated apparatus in Fig. 1, the feed, which may be a petroleum distillate or any other mixture of substances separable into fractions of different volatilities, is supplied to the column 1 by means of pipe 2 and manifold 3. The feed may be introduced cold, heated, partially or entirely vaporized, for example, by application of direct heat or by passing through one or several heat exchangers 25. Vapors within the column pass countercurrently to and in contact and heat exchange relationship with the descending liquid. The ultimate vapor mixture at the top of the column is withdrawn by means of pipe 5, condensed in the condenser 6, and is collected in the surge tank 8 from which it is continuously withdrawn by pump 12 and divided in a constant ratio by valves 13*a* and 13*b* between the reflux pipe 15 and the liquid withdrawal pipe 14.

The liquid product formed within the column 1 is collected at the bottom of the column and may be withdrawn through pipe 4 at a rate regulated by valve 21 responsive to level controller 20, after being passed through the heat exchanger 25, or by-passed by means of pipe 31.

A substantially constant pressure is automatically maintained in the system by the controller 16, which actuates valve 22, controlling the flow of cooling medium in pipe 7, or valve 30, controlling the flow of vapors by-passed around the condenser by pipe 29, or again by regulating the steam flowing through a coil 32 which may be installed for the purpose in the surge tank 8. Controller 16 may be made responsive to the pressure variations in column 1, or, if desired, in tank 8.

Supposing that the fractionating unit of Fig. 1 is operated for a specification top product, and the operation is to be maintained for an optimum yield, and assuming that the fractionating process has reached the point of stable operation, a certain rate of feed is then maintained through the valve 11, corresponding to a certain liquid level in tank 8. If now the composition of the feed changes so that the content of volatile components increases, an increased amount of material will reach the reflux condenser 6. The amount of cooling medium going to this reflux condenser remaining constant, this apparatus will fail to condense all the vapors and, as a first result, the pressure on the system will show a tendency to rise. As a consequence, the pressure controller 16 will open the regulating valve 22 and thus admit an increased amount of cooling medium to the reflux condenser until such an increase has been realized that the former pressure is reestablished. As the increased amount of vapors will now again be totally condensed in the reflux condenser, the amount of liquid flowing into tank 8 will increase and since a constant amount is removed from this tank by means of pump 12, the liquid level in tank 8 will rise. This in turn will cause the liquid level controller 10 to act upon the feed regulating valve 11 in a manner which will reduce the rate of feed to the column. In

this manner, the controlled liquid level in tank 8, which depends upon the total amount of overhead, i. e. the sum of the reflux and of the top product, is used for regulating the rate of feed and therefore the load on the column. The yield may be simultaneously controlled through the valves 13a and 13b, keeping a constant ratio between the reflux and the top product withdrawn.

In this manner it is possible to obtain:

(a) A constancy of overhead (reflux plus top product) which assures a continuous operation at any desired constant load, including the optimum;

(b) A constancy in the ratio of reflux to top product which in turn assures a constancy in the degree of fractionation;

(c) A constancy in the amount of the desired top product;

(d) A continuous regulation of the rate of feed in accordance with both the desired load and the desired yields;

(e) A constancy of working pressure which, in conjunction with the temperature control 17 or 19, assures a constancy of the specified properties of the top product by means of valve 23 regulating the heat input to the column.

A similar combination of either automatic or manual control and regulation would adjust the rate of feed in case the content of volatile components should diminish. The receding level in tank 8 would then cause the opening of the valve 11 to admit more fluid, while all other features of control and regulation would proceed in a manner insuring a stable operation of the system.

When the fractionating unit is operated for a bottom product of specified properties, a somewhat different arrangement of controlling and regulating means may be used, such, for example, as illustrated in Fig. 2. Instead of means 17 and/or 19, the temperature control means 17B may be used to control the specified properties of the bottom product in cooperation with the constant pressure control means 16.

Tank 8 may in this case be replaced by a standpipe 8B, which has a solid partition 26 separating it into two liquid-tight compartments, communicating by means of a pipe 27, in which a constant flow is maintained, for instance, by a master-controller or liquid head controller, 10B actuating valve 28. The same pressure is maintained in both compartments by means of a pipe 12B connecting their vapor spaces. The purpose of this arrangement is to use the variable liquid head in the upper section of tank 8B to actuate the master-controller 10B, which in turn regulates the flow of the reflux by means of valve 24, the flow of the feed in pipe 2 by means of valve 11, and the flow of the top product in pipe 27 by means of valve 28. The master controller is connected to both the liquid and the vapor phases in the standpipe 8B, so that it is not influenced by variations of the total pressure in the system but only by variations in the liquid head.

Assuming then, as in the first case, that the amount of volatile components in the feed to column 1 increases, the pressure of the system will rise, and the pressure controller 16 will admit more cooling medium to the condenser 6, thus reestablishing the pressure but increasing the amount of condensate. The liquid head in the upper section of standpipe 8B will accordingly rise, nominally tending to increase both the flow of the top product in pipe 27 and the flow of the reflux in pipe 15B. However, the action of the increased head on the master controller or liquid head controller 10B is such that the latter will reduce the opening in both valve 28 and valve 24, maintaining both flows constant at the desired rates. Simultaneously the master controller will reduce the rate of admission of feed to column 1 by partially closing the valve 11, thus ultimately decreasing the liquid head in standpipe 8B and bringing the whole system back to normal operation. An opposite automatic adjustment will take place if the amount of the lighter components in the feed decreases since a fall of the level in standpipe 8 will in that case actuate the master controller to open valves 24, 28 and 11 wider, thus keeping the flow of the reflux and of the top product constant and increasing the rate of feed admission.

It is obvious although I have described the use of both a liquid level controller and a static head controller as illustrated by Figs. 1 and 2 to regulate the load upon the column or the amount of yield, these apparatus may be used interchangeably whether the system is operated for a top or a bottom product. As an example, a system comprising a standpipe 8B, but operated for a top product is shown in Fig. 3. The same result might also be achieved by means of registration and manual control at these various points.

While in the two examples given the top product is removed as liquid, it will be realized that the method applies equally well if it should prove desirable to remove it in vapor form.

Again, while the method of feed regulation has been used simultaneously with the control of the temperature regulating the heat input into the reboiler 18, and with control of the reflux ratio, it should be understood that these items are distinctly separate and may be used individually one without the other.

Moreover, this system of control and regulation is adapted not only to operations at atmospheric and higher pressures, but may readily be used at any pressure including any degree of vacuum.

It is obvious, that these principles of control may also be applied to a whole system comprising several fractionating units in series and/or in parallel.

I claim as my invention:

1. In a process of fractional distillation, the steps of counterflowing in a fractionating zone a vaporized hydrocarbon mixture and a reflux formed in a condensing zone, automatically maintaining a constant pressure in the fractionating zone by regulating the rate of indirect cooling in the condensing zone solely as a function of the pressure in the fractionating zone, automatically regulating the flow of the material being fractionated to the fractionating zone as an inverse function of the rate of flow of the condensate formed in the condensing zone, automatically maintaining a constant reflux ratio, and automatically regulating the heat input to the fractionating zone as an inverse function of the temperature at the top of said fractionating zone.

2. In a continuous process of fractional distillation wherein a mixture of several components is separated into vaporized top and liquid bottom products, the steps of condensing the vaporized top product substantially under the pressure of the distillation zone, accumulating the condensate in a pool of restricted cross section and substantial depth, returning one portion

of the accumulated condensate to the top of the distillation zone under a hydraulic head of said pool, transferring another portion of the condensate also under a hydraulic head of said pool to an accumulating zone maintained under distillation pressure, maintaining said pressure constant by automatically regulating the rate of condensation of the vaporized top product as a direct function of said pressure, thereby varying the depth of the condensate pool, within a narrow range, counteracting the variations in the depths of the condensate pool by regulating the rate of condensate transfer to the accumulating zone, maintaining the rate of reflux constant irrespective of the variations in the depth of the pool, regulating the rate of feed to the distillation zone inversely to variations in the depth of the condensate pool, and automatically regulating the heat input to the fractionating zone as an inverse function of the temperature of said fractionating zone.

PETRUS JURJEN ROELFSEMA.