Oct. 21, 1941. T. O. WILTON 2,260,072

DISTILLATION AND FRACTIONATION OF MIXTURES OF HYDROCARBONS

Filed March 25, 1939 2 Sheets-Sheet 1

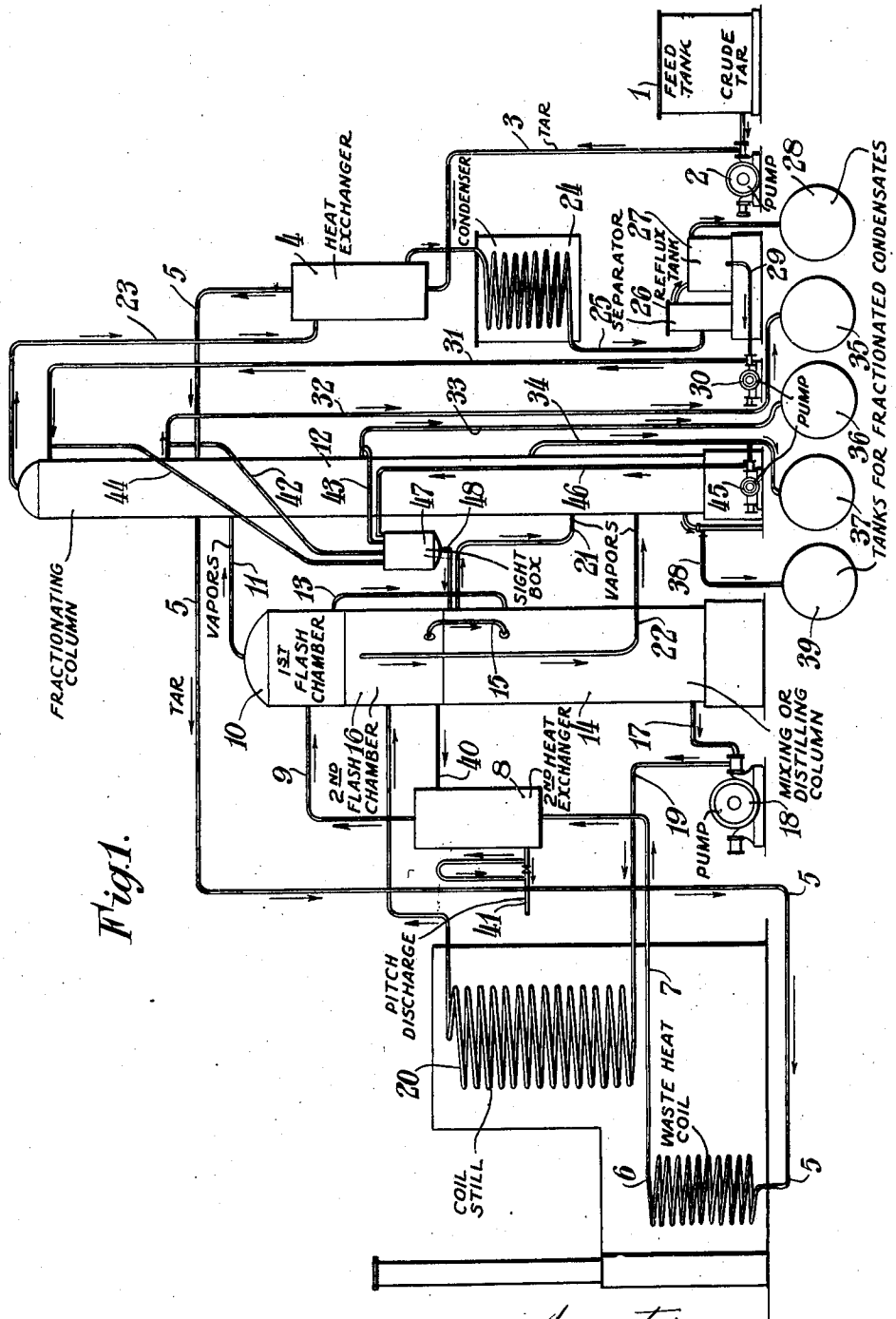


Fig. 1.

Inventor:
Thomas Overton Wilton
by Fraser, Myers & Manley
attorneys

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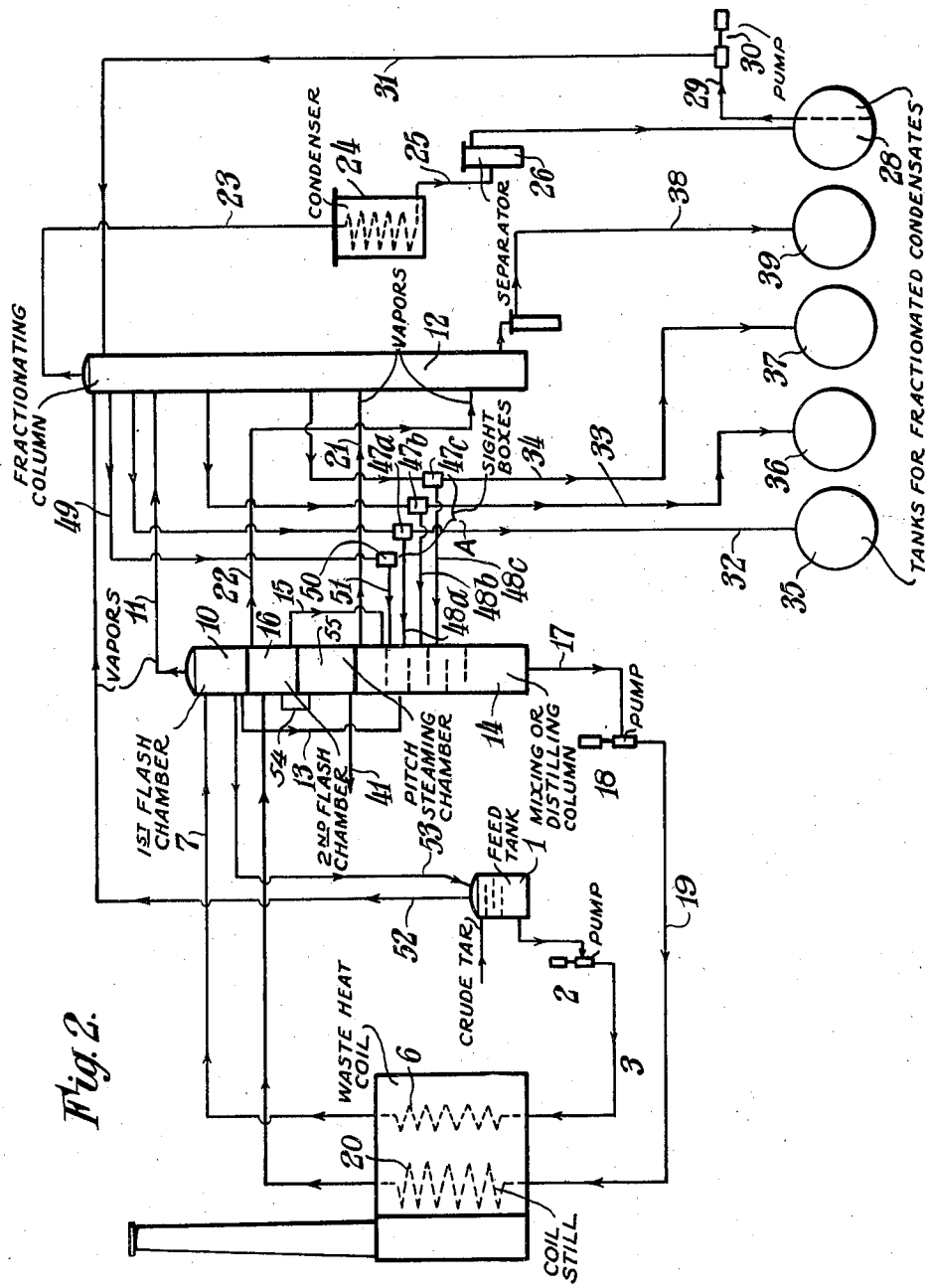


Fig. 2.

Inventor:
Thomas Ouston Wilton
by Fraser, Myers & Manley
attorneys

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DISTILLATION AND FRACTIONATION OF MIXTURES OF HYDROCARBONS

Thomas Owston Wilton, London, England

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In Germany May 16, 1938

2 Claims. (Cl. 196—73)

The present invention has for its object a process for distillation and fractionation of mixtures of hydrocarbons and apparatus for carrying out the said process.

In distillation and fractionation plant systems, and particularly in plants for the dehydration, distillation and fractionation of a mixture of liquid hydrocarbons such as coal tar, the vaporizable constituents, of various boiling points, are vaporized and the vapours are introduced in a fractionating column, generally of the type known as the bubble cap column, the fractions in the liquid phase being collected in side streams at various points of the fractionating column, in the usual manner, the vapours being admitted into the fractionation column either at one point only of the column, or at several points along the length of the column.

In practice, however, it is found that when this mode of procedure is applied to a process embodying a recirculation of materials from which low-boiling point constituents have been separated, the flexibility of the plant, derived from the recirculation of the material in the plant, is reduced owing to the very principle on which a bubble cap column functions: if the load falls off on the distillation unit the amount of vapours passing to the bubble column is reduced, and the quantities of condensable liquids are reduced. This canont be corrected by the application of superheated steam, since this steam remains in the vapour phase along the whole length of the distillation column, and so the margin of flexibility of the plant in fractionation, is but about 25 per cent of the total capacity of the plant, whereas the re-circulating is flexible from zero to full load.

According to my invention, I provide a process for adjusting the rate of flow of the vapours in the fractionating column by separating measured quantities of the side streams from the fractionating column, with the aid of suitable mechanism such as sight boxes and valves, and returning such separated portions into the distilling column, either separately, or after mixing them by admitting them to a container having a single outlet conduit. In this manner, the correct quantity of vapours of different boiling points is provided, so as to maintain optimum conditions of working in the fractionating column. My invention consists therefore in applying the re-circulation principle to the fractions from the fractionating column.

With this arrangement, I obtain an absolute flexibility in the load on a unit plant from full load down to zero, which is a feature lacking in any other type of process.

For example, assuming a plant designed to distil 100 tons of tar from which 50 per cent of pitch would be obtained, the remainder, namely, 50 per cent, being vapours, the bubble cap column would be designed with a certain diameter in order that the vapours travel in this column with a speed of about 80 feet per minute, which is recognised as being the optimum speed in a fractionating column, that is, the speed giving the finest cuts.

Let us now assume that it is required to work the same plant with a different kind of tar, giving 60 per cent of pitch of similar melting point to that of the first pitch, and therefore 40 per cent of vapours only, and that the same quantity, namely, 100 tons, is to be dealt with. Owing to the smaller amount of vapours generated, the speed in the bubble cap column will be reduced to about 64 feet per minute. It is a well known fact that the speed of the vapours in the bubble cap column determines the fineness of the cuts produced, and, therefore, the new conditions will not be so favourable to the production of fine cuts, and it would not be possible to get as fine cuts with the new tar as with the first one. According to my invention, 25 per cent of the condensates formed in the bubble cap column are sent back into the distilling column so as to form artificially in this column an additional quantity of vapours which will maintain the speed in the bubble cap column at the optimum figure of 80 feet per minute.

According to one embodiment of my invention, I provide a sight box on each side stream pipe, and I arrange for the sight boxes to be situated on a platform between the bubble cap column and the distilling column, and I provide an adjustable valve to each sight box, enabling an adjustable quantity of condensate from each sight box to be returned into the distilling column. In this manner, the speed of flow of the gases in the bubble cap column can be either increased or reduced, as may be desired, according to the amount of vapours available. When the amount of fuel required to bring about this adjustment is calculated, it is found that it is negligible for this reason, that the condensates coming from the bubble cap column are very nearly at their boiling point temperatures, or volatilization points, and are therefore vapourized again with a negligible expenditure of heat.

The advantage of the arrangement just described consists in the fact that a tar distiller who

receives various kinds of tars, and wishes the throughput of his plant to be maintained, can be assured of getting the same quality of cuts from the bubble cap column whatever the pitch content of the tar may be, and also the same pitch hardness, constantly.

The process and apparatus lend themselves to the distillation and fractionation of mixtures of hydrocarbons of different natures; for the purpose of illustration I will describe the application of the invention to the distillation and fractionation of coal tar.

The accompanying drawings represent, diagrammatically, as examples only, apparatus adapted for carrying out the herein-described process in accordance with the disclosed invention, Fig. 1 illustrating one exemplification of such apparatus and Fig. 2 a modification thereof.

Referring to the apparatus shown in Fig. 1, the crude material from feed tank **1** is fed by pump **2**, to and through pipe **3**, the heat exchanger **4**, pipe **5**, waste heat coil **6**, pipe **7**, second heat exchanger **8** and pipe **9** to a first flash chamber **10** where low boiling point vapours are separated and are led by pipe **11** to the upper portion of the fractionating column **12**. The first residues flow by pipe **13** from the first flash chamber to a mixing or distilling column **14**, where they mix with hotter second residues flowing through the pipe **15** from a second flash chamber **16**, the mixture of residues being conducted by pipe **17** to pump **18**, by which they are caused to flow through pipe **19**, and coil still **20** to the second flash chamber **16**.

The vapours of intermediate boiling point characteristics separated in the mixing chamber **14** are led by pipe **21** to an intermediate zone of the fractionating column **12**, while the vapours of high boiling point characteristics, separated in the second flash chamber **16**, are led by pipe **22** to a lower level of the fractionating column **12**.

The light oils in the vapour phase flow from the fractionating column **12** through the pipe **23**, and heat exchanger **4** to the condenser **24**, from which the condensate, crude benzole, is conducted through pipe **25**, separator **26**, and reflux tank **27** into a storage tank **28**. Some of the crude benzole which is led into the tank **27**, is withdrawn from said tank through a pipe **29**, by a pump **30**, and is thence returned by pipe **31** to the top of the fractionating column **12** for the purpose of controlling the temperature of the vapours escaping by pipe **23**.

Fractions (e. g. heavy naphtha, naphthalene oil and creosote) flow by pipes **32**, **33** and **34** to storage tanks **35**, **36** and **37**, respectively, while anthracene oil collecting at the bottom of the fractionating column flows by pipe **38** to the storage tank **39**. The final residue or pitch from the second flash chamber **16** flows by pipe **40** through the heat exchanger **8** and is discharged at **41**.

In accordance with the invention, a part of the side streams are tapped by pipes **42** and **43**, and led to a single sight box **47**, together with a part of the reflux oil, by pipe **44**. If desired, a part of the high boiling point fraction is withdrawn from pipe **34** by a pump **45** and is also led by pipe **46** to the sight box **47**, the arrangement being such that the several materials flowing into the sight box **47** are in the requisite proportions to keep the fractionating column under working conditions as uniform as possible. The mixture flows from the sight box **47** by pipe **48** into the distilling column **14** and thence is reintroduced in the cycle, the vapours of different boiling points being again separated and led by pipes **21** and **22**, respectively, to the fractionating column, from which they issue in the liquid phase as before.

For example, if when the plant is working with a certain crude material, the fractionating column is so adjusted that, for optimum working conditions, the quantities of materials flowing in pipes **25**, **32**, **33** and **34**, respectively, are 1%, 5%, 10% and 15%, and a different crude material is charged into the tank **1**, yielding 3% of heavy naphtha instead of 5%, which would upset the optimum conditions obtaining in the fractionating column, ⅔ of the 3% of heavy naphtha which would at first be available in the fractionating column on starting the plant, that is, 2% of such material, would be returned through the sight box to the distilling part of the system which, with the 3% being produced from newly added crude stock, would make up the required 5% of such material. This would at first leave but 1% of heavy naphtha to be withdrawn. It will be apparent, however, that, as the recirculation continues, the amount of crude naphtha which would be available at the level of the fractionating tower from which that product is to be withdrawn would gradually increase until 3% of heavy naphtha, that is, the amount produced from the crude stock being constantly fed into the distilling part of the system, could be withdrawn without interfering with the required flow of 5% of such material in the pipe leading from the distilling apparatus to the fractionating tower.

In like manner the working conditions may be modified respecting the proportions of the other side streams.

Should the crude material charged into the tank **1** yield less than half of the amount of any particular distillate than that needed for optimum working conditions, as, for example, say 2% of heavy naphtha when the apparatus is so adjusted as to require 5% to give the best results, it would be necessary, on first starting the plant, to return the entire amount first available at the pipe **32** and to permit the recirculation to be augmented until 2%, that is, an amount equal to that being added as a result of the crude material being constantly fed into the system, could be withdrawn for storage without reducing the amount being fed to the fractionating column below the desired 5%.

Referring now to the apparatus shown in Fig. 2, which represents a modification of the arrangement shown in Fig. 1, the side streams from the fractionating column **12** are brought together at a point A conveniently situated between the distilling column and the fractionating column, and the side stream pipes **32**, **33**, **34** are provided respectively with sight boxes **47***a*, **47***b*, **47***c* from which pipes **48***a*, **48***b*, **48***c* lead adjustable quantities of fractionated condensates into the distilling column.

If desired an adjustable portion of the light oil of the top section of the fractionating column may be led by pipe **49** to a sight box **50** and thence by pipe **51** into the distilling column.

In the apparatus shown, part of the residues from the first flash chamber **10** are led back by pipe **53** into the feed tank **1**, in which some vapours of low boiling point are liberated and sent by pipe **52** into the fractionating column **12** near the top thereof, and the rest of the residues are led by pipe **13** to the distilling column **14**.

Instead of passing the final residues or pitch to be withdrawn from the second flash chamber 16 through pipe 40 to a heat exchanger 8, as in the first form of the invention hereinbefore described, this product may be conducted by pipe 54 to a pitch steaming chamber 55, from which it may be withdrawn through discharge pipe 41. The remainder of the residues to be withdrawn from the second flash chamber may, as in the case of the first described form of the invention, be conducted to the distilling column 14 by pipe 15. There are therefore four groups of vapours having different boiling points, entering the fractionating column at four different points along its length. Two of these are liberated by the waste heat of the combustion gases of the furnace and the two others by the heat of the furnace itself.

The modified form of apparatus, adapted for use in practicing the process embodying the herein-disclosed invention, which is illustrated by Fig. 2, includes many elements which have not been specifically referred to. Such parts, however, are identified by reference characters conforming with those by which corresponding elements of the apparatus illustrated by Fig. 1 have been designated, and, since such corresponding elements of the two forms of mechanism which have been disclosed are substantially identical, further specific description would appear to be unnecessary.

By means of the arrangement described above, an absolute flexibility is obtained for the plant, from full load to zero.

What I claim is:

1. A process adapted for use in the fractional distillation of hydrocarbon compounds, which consists in continuously subjecting increments of the compound to a treatment of distillation in the first stage of which they will be commingled in a heat transferring relation with residues of material previously subjected to treatment administered pursuant to a second stage and of a temperature such as to vaporize components of relatively low boiling point characteristics; separating the vaporized components from the commingled residues and, as the said second stage of the treatment, continuously withdrawing residues from the said commingled mass and heating them to a temperature such as to vaporize components of boiling point characteristics materially greater than those vaporized pursuant to the first stage; separating the components vaporized pursuant to the second stage from the residues; continuously conducting a part of said residues to the commingled mass being treated pursuant to the first stage for use as the heating agent thereof and, as a third stage, conducting the remainder of the residues resulting from the treatment pursuant to the second stage to and causing them to be commingled in a heat transferring relationship with residues of material previously subjected to treatment administered pursuant to a fourth stage and of a temperature such as to vaporize components of boiling point characteristics materially greater than those vaporized pursuant to the second stage; separating the components vaporized pursuant to the third stage; constantly withdrawing residues from the commingled mass being subjected to treatment pursuant to the third stage and, pursuant to the said fourth stage, heating them to a temperature of a degree such as to vaporize components of boiling point characteristics materially greater than those vaporized pursuant to the third stage; separating the components vaporized pursuant to the fourth stage; conducting the separated vapors to different levels of a fractionating tower, graded from an uppermost level to receive the vapors of lowest boiling point characteristics to a lowermost level to receive those of highest boiling point characteristics; condensing fractional components of the compound differentiated by materially different boiling point characteristics at different levels of the tower; withdrawing said condensates from the different levels for storage as finished products; and effecting a degree of control over the rate of flow of vapors from one level to another of the tower and of the relative density of vapors at different levels thereof, by separating a part of the condensate drawn from one of the levels of the tower, adding the said separated condensate to material being subjected to the treatment of distillation and thus causing it to be revaporized and returned to the tower with other components being vaporized and conducted to the tower as a result of such treatment.

2. The process defined by claim 1, further characterized in that portions of the condensates withdrawn from a plurality of the levels of the tower are separated and added to material being subjected to the multi-stage treatment of distillation, so as to be caused to be revaporized and returned to different levels of the tower with other components being vaporized and conducted to the tower as a result of such treatment.

THOMAS OWSTON WILTON.