

Oct. 17, 1939.

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2,176,498

VACUUM RECTIFYING COLUMN

Filed July 2, 1937

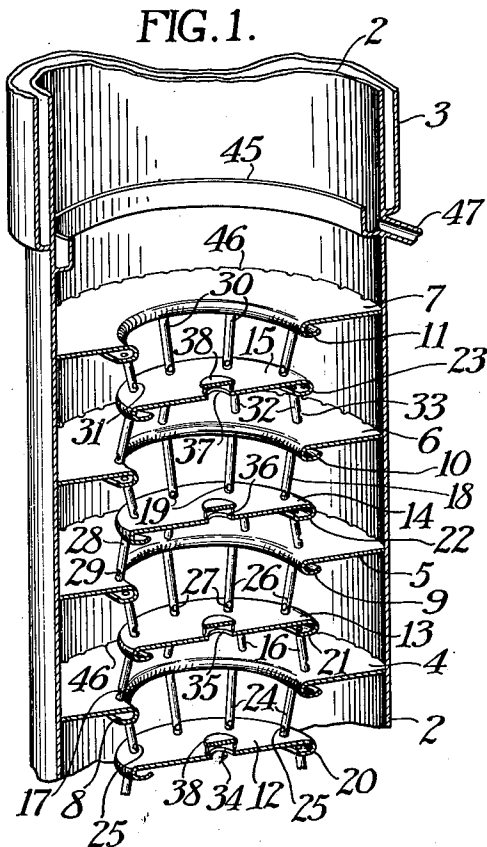
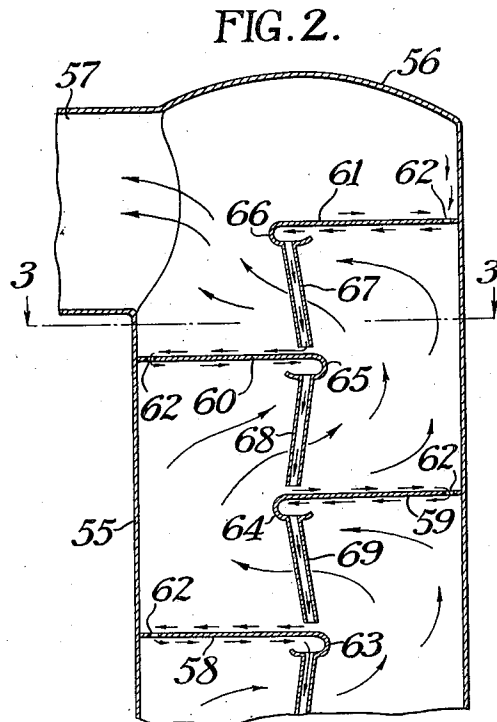
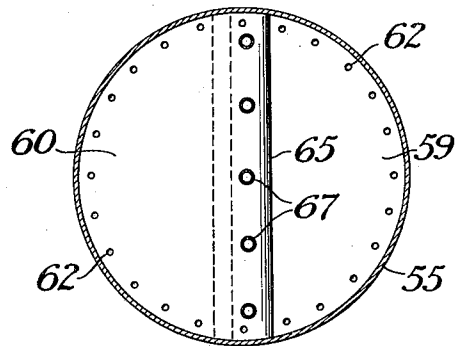
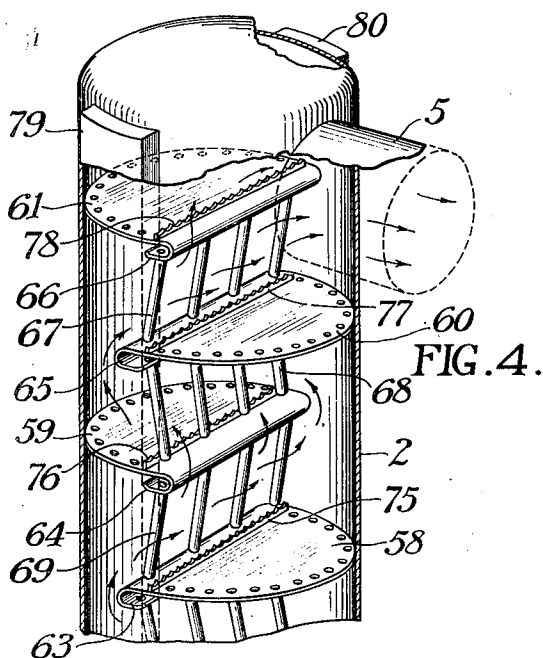


FIG. 3.



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2,176,498

VACUUM RECTIFYING COLUMN

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Application July 2, 1937, Serial No. 151,715
In Great Britain July 6, 1936

9 Claims. (Cl. 261—114)

This invention relates to improvements in the fractionation or rectification of vapors under vacuum and more particularly to improvements in vacuum rectifying columns.

The conventional rectifying column for use at ordinary pressures is filled with a series of bubble cap plates which ensures thorough washing of the reflux by the ascending vapors. This construction is known to be unsatisfactory for vacuum and particularly high vacuum fractionation, due to the high resistance to the flow of vapors through the column. Relatively high pressures therefore prevail in the still and column, even though high capacity evacuating pumps are employed. To avoid these disadvantages, it has become common practice to employ plates which are so designed and located that the ascending vapors are substantially unconstricted in passing through the column. This construction avoids "pressure drop" to a considerable extent, but does not avoid other difficulties. The vapor velocity under vacuum conditions is so high that in a column of this type condensate is swept along with the vapor stream and is, therefore, substantially prevented from freely flowing counter-current thereto, as is necessary to obtain effective interchange between the two. Poor fractionation and flooding of the column, therefore, takes place. This is especially troublesome at low pressures, such as at .001 to 50 mm. where vapor velocities are extremely high.

This invention has for its object to overcome the above deficiencies of vacuum, and particularly high vacuum, rectification. Another object is to provide an improved method of vacuum rectification. A further object is to provide method and means whereby the adverse effect of the high velocity vapor streams is substantially avoided. A still further object is to provide an improved vacuum rectifying column. Other objects will become apparent from the following detailed description.

These and other objects are accomplished by the herein described invention, which in general comprises collecting the condensate from an upper plate and passing it to the next lower plate in the column while protecting it from the action of the vapor stream.

To facilitate the description of my invention, reference is made to the accompanying drawing in which:

Fig. 1 is a vertical section in perspective of one type of fractionating column embodying the principles of my invention;

Fig. 2 is a vertical section of a modification of

the column shown in Fig. 1, being provided with semi-circular plates;

Fig. 3 is a horizontal section taken on line 3—3 of Fig. 2;

Fig. 4 is a vertical section in perspective of apparatus substantially the same as that illustrated in Fig. 2, but being provided with external heating elements and means for aiding in the even distribution of condensate over the surface of the plates.

Referring to Fig. 1, reference numeral 2 designates a cylindrical column which is connected at the base to a source of vapor and which is provided at the top with a jacket 3 through which cooling fluid is circulated in order to cool the upper walls of the main casing 2. Numerals 4, 5, 6 and 7 designate circular plates supported by and fixed to the walls of the cylindrical casing 2 in such a manner that they are approximately horizontal. These plates are provided with a central opening, the inside edge of which is turned under to form gutters 8, 9, 10 and 11, respectively. Numerals 12, 13, 14 and 15 designate circular plates which approximately correspond in diameter to the diameter of the central opening in plates 4—7. The outside edges of plates 12—15 are turned under to form gutters 20, 21, 22 and 23. Plate 12 is rigidly supported in the central position, illustrated by a series of pipes 24, which communicate with gutter 8 and are rigidly fixed thereto and which are also rigidly fixed to the upper surface of plate 12. These pipes are provided with openings 25 at a position just above the surface of plate 12, located upon the side of the pipe facing towards the center of the column. Plate 13 is similarly supported by a series of pipes 26 located about its periphery, which communicate with the gutter 9 and are provided with openings 27 at the base thereof. Another series of conduits 16, having openings 17 in the base thereof, connect to gutter 21 and deliver liquid therein onto the upper surface of plate 4. Plate 14 is supported by a similar series of conduits 18, which communicate with gutter 10 and deliver liquid through openings 19 onto the upper surface of plate 14, and another set of lower conduits 28, which deliver liquid from gutter 22 onto the upper surface of plate 5 through openings 29. Plate 15 is supported in substantially the same way by conduits 30 communicating with gutter 11 and having openings 31 in the base thereof, and by a similar series of lower conduits 32 provided with openings 33 which connect gutter 23 and the upper surface of plate 6. Plates 12 to 15 are provided with centrally lo-

cated openings 34—37, over each of which is suspended a cap 38. The outside peripheries of plates 4—7 are notched at 46 to provide openings for the flow of condensate from the upper side, down onto the lower side of the plate. Numeral 45 designates an annular gutter which is connected to a withdrawal conduit 47.

Referring to Figs. 2 and 3, numeral 55 designates the casing of a fractionating or rectifying column which is provided with a dome 56 and which is connected to conduit 57 at its upper end. Numerals 58, 59, 60 and 61 designate approximately horizontal semi-circular plates rigidly fixed to the walls of the main casing 55 and which are provided with a series of openings 62 about the periphery which are in close proximity to the wall of casing 55. Numerals 63, 64, 65 and 66 designate gutters formed at the free edge of the semi-circular plates 58—61 by turning the edge thereof in the manner illustrated. A plurality of conduits 67 communicate with gutter 66 and terminate a short distance above the surface of plates 60. A similar series of conduits 68 communicate with gutter 65 and terminate a short distance above the surface of plate 69. Another similar set of conduits 69 communicate with gutter 64 and terminate a short distance above the surface of plate 58.

Referring to Fig. 4, numerals 75, 76, 77 and 78 designate a serrated blade which acts as a weir in evenly distributing liquid over the surface of plates 58, 59, 60 and 61. Numerals 79 and 80 designate external heating units, preferably electrical heating pads, which are located on diametrically opposite sides of the column 2 and in positions corresponding to the location of the ends of the gutters 63, 64, 65 and 66.

In operating the apparatus of Fig. 1, cooling fluid is circulated in the space between the upper walls of column 2 and jacket 3. The upper end of the column 2 is connected to vacuum pumps having sufficient capacity to evacuate the system to the desired low pressure. The base of the column is connected to the vapor source which, of course, must be gas tight. The vapors ascend through the column passing into the space between plate 12 and plate 4. In passing through this space, the vapors necessarily must flow toward the axis of the column. The vapors then are reversed in their path of flow and pass between the plate 4 and central plate 13, the vapors passing toward the outside walls of the casing 2. This repeated reversal of the direction of flow of the vapors takes place until they eventually pass through the central opening in the top plate 7 and are condensed upon the cold upper walls of column 2. Condensate flows into gutter 45 and is withdrawn through conduit 47.

Less volatile constituents condense upon the various plates as the vapors pass in the zigzag path through the column. Condensate from plate 7 flows to the openings 46 about the periphery thereof, down and onto the under surface of the plate. The vapors ascending the column at this point are flushing or brushing the under surface of plate 7 in a direction towards the axis of the column. Therefore, liquid passing through the openings 46 is caused to flow into the gutter 11 by this action of the vapors. At the same time, interchange between the film of condensate and the vapors take place and volatile components thereof are again vaporized and carried along with the vapor stream. The washed liquid collecting in gutter 11 flows down through conduits 30 onto the upper surface of plate 15.

The vapors in contact with the upper surface of this plate are also passing in a direction toward the axis of the column and the liquid flowing from these conduits is, therefore, blown towards the opening 37 and passes therethrough onto the under surface of plate 15. The ascending vapors in contact with the under surface of plate 15 are passing at high velocity towards the outer walls of the column and the liquid passing through the opening 37 is, therefore, forced or blown into gutter 23. At the same time, interchange takes place between the film of condensate and the vapor phase as explained above. Liquid collecting in gutter 23 then passes through conduits 32 onto the upper surface of plate 6, is blown over to the wall of casing 2, passes through the openings 46 onto the under surface of plate 6 and is then blown into gutter 10. Substantially the same action takes place on each of the sets of plates down through the column.

It is apparent that, by the construction illustrated, the high velocity vapors are caused to pass into intimate contact with a large area of condensate and that effective interchange takes place in its passage through the column. It is also apparent that washing of the condensate in an upward direction is avoided by passing it in protected paths in a downward direction at intervals while intermittently exposing it to the washing action of the vapor. The high velocity vapors are also caused to help force the liquids in the downward direction instead of forcing them in an upward direction as is usual with conventional vacuum column construction.

In operating the apparatus of Fig. 3, conduit 57 is connected to a high admittance condenser which is in turn connected by a large diameter conduit to a suitable vacuum pump. The lower end of the column is connected to a source of vapor such as the pot of a still. The ascending vapors pass into contact with the bottom surface of plate 58 and are forced to pass to the diametrically opposite side of the column, as illustrated by the long arrows. The vapors then pass along the bottom surface of plate 59 and are forced to pass to the opposite side of the column. In this way, the vapors take a zigzag course up through the column and eventually pass into the conduit 57 and thence into the condenser. Condensate collecting upon the top surface of plate 61 flows through the opening 62, along the bottom of the plate 61, where the force of the vapors blows it as a thin film into gutter 66. The direction of flow of condensate is shown by the short arrows. The liquid then flows through conduits 67 onto the upper surface of plate 60, where it is blown by the action of the vapors over into the holes 62 and passes down onto the bottom surface of plate 60 where the action of the vapors causes it to flow as a film into gutter 65. Substantially the same action takes place on plates 59 and 58. The condensate is, therefore, caused to flow both by gravity and by the dynamic action of the vapors in a downward direction through the column while effective interchange between large areas of the condensate and large volumes of the vapors takes place. Due to the wide spacing between the plates of all of the figures, slight resistance to the flow of vapors is offered.

The operation of the apparatus of Fig. 4 is substantially the same as that of Fig. 2 except that heating elements 79 and 80 cause vaporization of condensate collecting upon the walls of the column in their immediate vicinity. Such vaporization is advantageous, since the high velocity

of the vapors tends to blow the condensate around to the area corresponding to that covered by the heaters. This condensate then flows in a zigzag course down the walls of the column in this vicinity without being passed over the plates and, therefore, without effective interchange with the vapors. Heating of this area causes any such condensate collecting in this zone to be vaporized and passed with the main vapor stream into interchange with the condensate. The elements 75, 76, 77 and 78 cause even distribution of condensate over the entire surface of the plates 58 to 61.

Many changes and variations can be made in the above described apparatus, without departing from the spirit or scope of my invention. For instance, the entire outside surface of the column may be jacketed to provide for the circulation of a cooling or heating fluid. Also, certain sections of the column may be jacketed to enable the control of the temperature of any particular zone or area in the column. It is also desirable in many cases to provide part or all of the column with an insulating jacket or lagging to prevent heat losses and thus avoid the necessity of adding or subtracting heat. The column will be heated by vapors to an extent depending upon the rate of distillation which is, in turn, controlled by the temperature of the still. Heating of the column will not usually be necessary except with very low vapor pressure compounds or with very low rates of distillation. The number of plates employed depends upon the degree of separation required and the rate of distillation and can be varied within wide limits to suit these requirements. The plates may slant slightly upwards or downwards, but approximately horizontal plates are preferred, since they increase the rate of downward flow caused by the action of the vapors. If it is desired to fractionate under molecular distillation conditions, the plates can be spaced in the tower at distances of less than the mean free path under the particular pressures employed.

It will be apparent that the base of the column can be connected to any well known type of vaporizing apparatus, such as an ordinary pot still. Also, the base of the column may be used as the vapor source in well known manner, by disposing a heating element therein and introducing the material to be vaporized in ordinary or preheated condition. Since materials which are usually distilled under vacuum conditions have low vapor pressures and are difficultly volatile, it is best to employ a flash vaporizer as a vapor source. These permit almost instantaneous vaporization of small amounts of the material and avoid considerable decomposition due to the fact that all of the material is not heated during the whole distillation period. A flash vaporizer in which small amounts of the material are caused to flow over a heated revolving surface by centrifugal force is particularly advantageous.

Since the velocities of vapors at pressures of below 100 mm. such as at .001 to 50 mm. are exceptionally high, little interchange between vapor and condensate takes place in towers of the prior art. My invention is, therefore, of particular value for fractionations carried out within this pressure range. It is especially intended for use with vapors entering the bottom of the column at pressures of .01 to 50 mm. and particularly .1 to 10 mm., and leaving at the top at pressures of .001 to 20 mm.

Due to the fact that the edges of the plates

are turned under to collect any condensate on the plate, all splashing and dropping is substantially avoided and this fact materially contributes to the avoidance of entrainment. Since the features of my invention permit the use of widely spaced plates which have been found to be necessary in vacuum, and particularly in high vacuum distillations, substantially all back pressure problems due to constriction of vapors are avoided. The outstanding advantage of the construction described is that obstruction to reverse passage is avoided, since the vapors are prevented from coming in contact with condensate at the time that it is flowing towards the base of the column. However, the vapors are contacted at intervals with the condensate while it is in the form of films of large surface area. During such contact the condensate travels in the same direction as the vapors and yet it is always being carried counter-current to the vapors by the gutters and conduits. However, even though the vapors and condensate travel in the same direction during contact, undesirable entrainment does not take place and the condensate is in fact helped to flow in a counter-current direction. At the same time very effective interchange takes place between the two.

What I claim is:

1. A vacuum fractionating column of the character described and comprising in combination, a plurality of approximately superimposed plates, which are widely spaced with respect to each other, which have a considerably smaller area than the cross sectional area of the column and which are adapted to retain condensate in the form of a film and means for conveying condensate from each of the plates to the next lowest plate while protecting it from the vapor stream.

2. A vacuum fractionating column comprising in combination, a main column casing, a plurality of approximately superimposed plates which are mounted inside the column casing, which are widely spaced with respect to each other, which have a considerably smaller area than the cross sectional area of the casing and which are adapted to retain condensate thereon in the form of a relatively thin film and means for collecting condensate from each of the plates and passing it to the surface of the next lowest plate while protecting it from the action of the high velocity vapor stream.

3. A vacuum fractionating column comprising in combination, a main column casing, a plurality of approximately horizontal superimposed plates which permit substantially unconstricted flow of vapor through the column which plates are so constructed that condensate is caused to flow thereover in the form of a thin film by contact with the moving vapor, and means located upon the plates for collecting condensate and passing it to the surface of the next lowest plate while protecting it from the high velocity vapor stream.

4. A vacuum fractionating column comprising in combination, a tower or main column casing, a plurality of approximately superimposed plates arranged therein which plates are so constructed that condensate is caused to flow over the top and bottom surface thereof in the form of a thin film by contact with the moving vapor, a vapor passage through or between the plates which offers slight resistance to the flow of vapors, and means for conveying condensate in a protected path from each upper plate to the next lowest plate.

5 5. A vacuum fractionating column comprising
in combination, an approximately vertical main
column, a plurality of approximately horizontal
plates spaced from each other in the column and
adapted to retain a film of condensate on the
top and bottom surfaces thereof, the plates cover-
ing only a portion of the cross sectional area
of the column and being so arranged in the col-
umn that the particular cross sectional area not
10 covered by one plate is covered by the plates
below and above it, means for collecting conden-
sate from each plate and means for conveying
the collected condensate in a protected path to
the next lowest plate.

15 6. A vacuum fractionating column of the char-
acter described and comprising a plurality of
approximately super-imposed plates, alternate
plates being staggered in relation to one an-
other so as to offer slight resistance to the flow
20 of vapors, means for allowing condensate to pass
in the form of a film over the top and bottom
surface of each plate substantially in the same
direction of flow as the vapors, and means for
conveying condensate from each of the plates to
25 the next lowest plate, while protecting it from
the vapor stream.

7. A vacuum fractionating column of the char-
acter described, which comprises in combination,
a main column casing, a plurality of approxi-
30 mately superimposed plates which offer slight
resistance to the flow of vapors which are adapt-
ed to retain a thin film of condensate on the top
and bottom surface thereof and which are so
disposed in the column that the vapors and con-
35 densate travel in the same direction when in con-
tact with the top and bottom surfaces thereof
and means for conveying condensate from each

of the plates to the next lowest plate, while pro-
tecting it from the vapor stream.

8. A vacuum fractionating column comprising
in combination an approximately vertical cylin-
drical casing, a plurality of semi-circular plates
5 having perforations about the circular edge
thereof disposed at intervals upon the internal
walls of the casing and in an approximately hori-
zontal position, each alternate plate being dis-
posed upon the opposite wall of the casing, col-
10 lecting gutters located under the straight edge
of each plate and conduits connecting each gut-
ter to the top straight edge of the next lowest
plate.

9. A vacuum fractionating column comprising
15 in combination, an approximately vertical col-
umn casing, a series of plates, having small per-
forations about their periphery and a large cen-
tral opening, disposed in a horizontal position
and at spaced intervals within the main casing,
20 collecting gutters at the edges of the central
openings of each plate and so located as to collect
liquid flowing along the bottom surfaces of the
plates, a second series of plates arranged in align-
ment with and corresponding in diameter to the
25 central opening in the first series of plates, and
which are disposed between each pair of the first
series of plates, collecting gutters at the edges
of the second series of plates and so located as
to collect liquid flowing along the bottom sur-
30 faces thereof, a plurality of conduits connecting
the gutters of the first series of plates with the
top edges of the next lowest of the second series
of plates to the top central edges of the next
lowest of the first series of plates.

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