

110

Feb. 20, 1945.

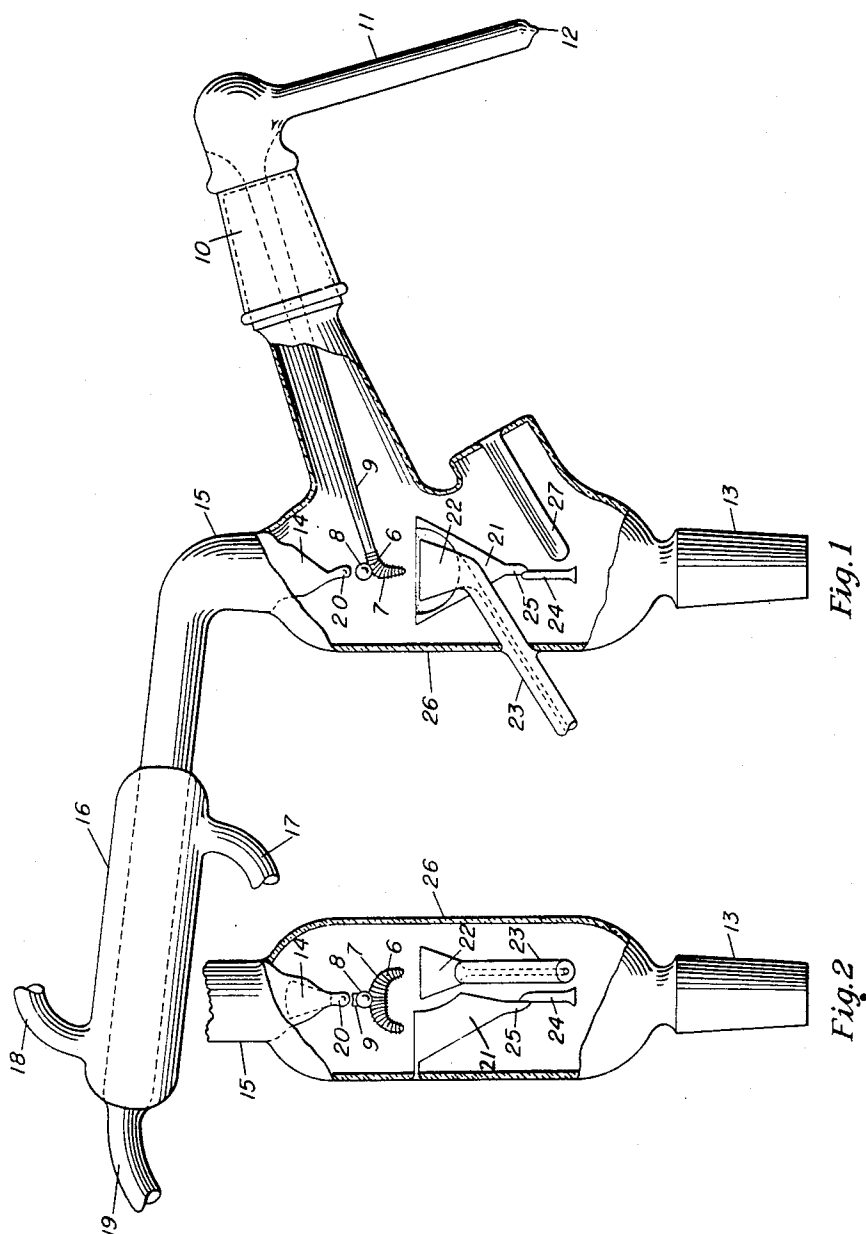
S. PALKIN ET AL

2,369,913

APPARATUS FOR PRECISION CONTROL OF FLOW DIVISION
FOR FRACTIONATING COLUMNS AND THE LIKE

Filed Aug. 11, 1943

4 Sheets-Sheet 1



Inventors
SA MUEL PALKIN, Deceased
By ROSE PALKIN, EXECUTRIX
STANLEY A. HALL
Attorneys
L. L. Richards
G. J. Kramer

Inventors
SAMUEL PALKIN, Deceased
By ROSE PALKIN, EXECUTRIX
STANLEY A. HALL
G. G. Kramer
L. L. Hildebrand
Attorneys

S. PALKIN ET AL

APPARATUS FOR PRECISION CONTROL OF FLOW DIVISION
FOR FRACTIONATING COLUMNS AND THE LIKE

FOR FRACTIONATING COLUMNS AND THE LIKE

Filed Aug. 11, 1943

4 Sheets-Sheet 3



23y

Inventors
SAMUEL PALKIN, Deceased
By ROSE PALKIN, EXECUTRIX
STANLEY A. HALL
A. J. Kramer
b. h. Richards
Attorneys

110

Feb. 20, 1945.

S. PALKIN ET AL
APPARATUS FOR PRECISION CONTROL OF FLOW DIVISION
FOR FRACTIONATING COLUMNS AND THE LIKE

2,369,913

Filed Aug. 11, 1943

4 Sheets-Sheet 4

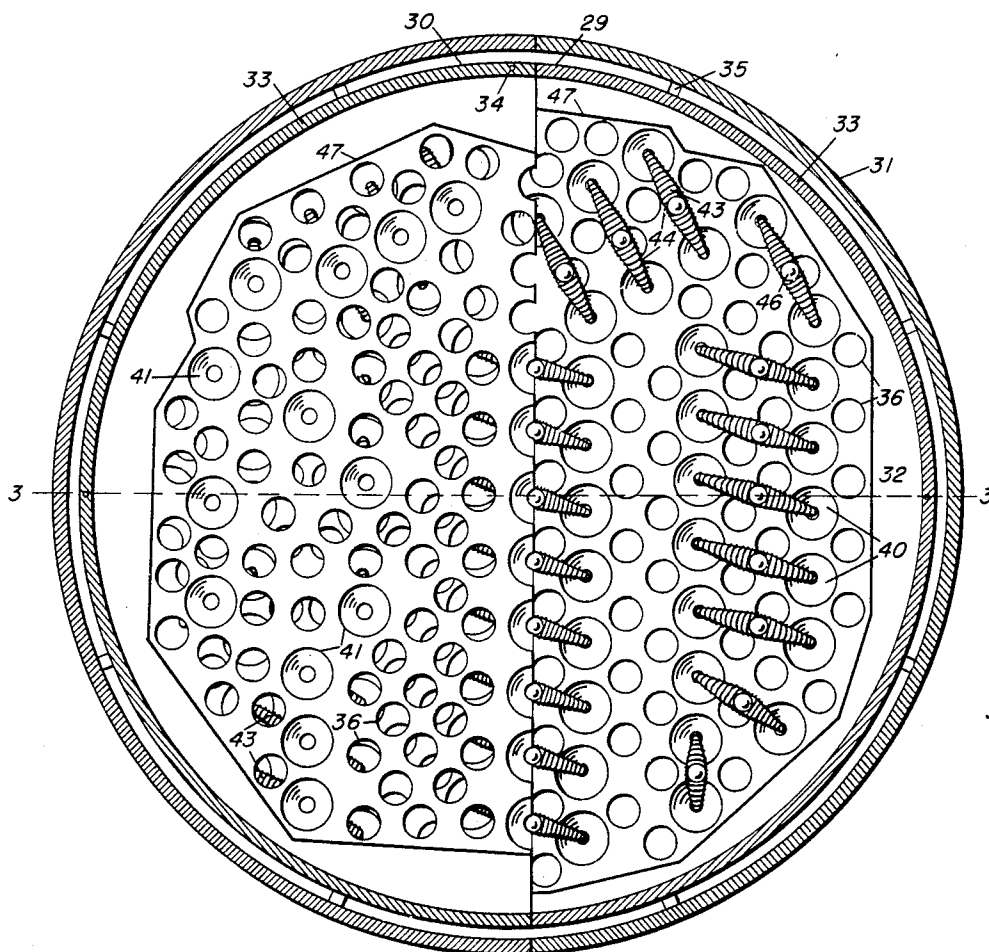


Fig. 5

Inventors
SAMUEL PALKIN, Deceased
By ROSE PALKIN, EXECUTRIX
STANLEY A. HALL
A. J. Kramer
L. L. Richards
Attorneys

UNITED STATES PATENT OFFICE

2,369,913

APPARATUS FOR PRECISION CONTROL OF
FLOW DIVISION FOR FRACTIONATING
COLUMNS AND THE LIKE

Samuel Palkin, deceased, late of Washington, D. C., by Rose Palkin, executrix, Wartrace, Tenn., and Stanley A. Hall, Mount Rainier, Md., assignors to the United States of America as represented by Claude R. Wickard, Secretary of Agriculture, and his successors in office

Application August 11, 1943, Serial No. 498,260

8 Claims. (Cl. 261-110)

(Granted under the act of March 3, 1883, as amended April 30, 1928; 370 O. G. 757)

This application is made under the act of March 3, 1883, as amended by the act of April 30, 1928, and the invention herein described, if patented, may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment to us of any royalty thereon.

Our invention relates to apparatus for effecting a division of liquid streams into component smaller streams and means of controlling the ratio of flow rates of such component streams with respect to one another and to the original stream.

In our pending application for patent, Serial No. 424,476, filed December 26, 1941, (now Patent No. 2,344,560, dated Mar. 21, 1944) covering multiple fractionating columns, scrubbing towers, and the like, certain column packings, concentric tubes and segmented rod-packings are described, for the proper wetting of which a reflux flow-dividing mechanism is necessary. That patent application is primarily concerned with means for overcoming certain difficulties in packed columns due to channeling (uneven wetting of the packing) by using a system of multiple columns instead of mass packing. Several types of such multiple column systems are described, and one flow-dividing mechanism for apportioning the liquid reflux to the individual column units is also described. This flow-dividing mechanism comprises a system of individual tubes and weirs fed by way of overflow of liquid from a common hydrostatic level. In the particular form of column packing described for use with this flow-dividing mechanism, the column units which give the highest separating efficiency are such as have relatively small diameter—one-half inch or thereabouts. Also, the reflux rate corresponding to peak efficiency for each column unit of small diameter comprising the multiple system, is relatively low—less than 100 cc. reflux per hour. Moreover, the efficiency of packed columns may be seriously impaired when the reflux rate per unit is allowed to exceed appreciably this low rate. Consequently, in a multiple column system, a means must be available (1) for the precise control of flow rates of small magnitude—less than 100 cc. reflux per hour, and (2) the control mechanism must be sufficiently simple and effective to permit divisibility of an incident stream into the large number of equal small streams, necessary to feed all the column units at the same rate.

Another instance in column operation, in

which accurate flow division constitutes an essential function, is in the control of "reflux ratio" (ratio of condensate returned to the column to that taken off as finished product). For this purpose, while only a single flow divider is necessary, the flow rates involved are of much smaller magnitude. For example, it may be necessary to divide an incident stream of 50 cc. per hour into two streams—one, 49 cc. per hour (return reflux) and another, 1 cc. per hour (product). So far as the present invention concerns applications to fractionating columns, the single flow divider unit (tilting arc form shown in Figures 1 and 2) is the form used for adjusting reflux ratio, while the multiple system of arcs (Figures 3 through 5) is used for adjusting feeding rates of reflux to a multiple column system.

While any number of simple flow-dividing devices are available for handling rapid streams (about 1000 cc. per hour) or over, as for example, a basin with a system of fine holes, such devices will be found in practice inoperable for flow rates below 50 cc. per hour, as holes of exceedingly fine bore and very great uniformity of bore would be necessary. Also, such fine bore and high uniformity of bore are not only in themselves difficult to obtain, but even when a series of such holes has once been made, the holes are highly susceptible to clogging by particles and sensitive to change by corrosion, which renders such a system of division undependable. Moreover, even if it were possible to prevent clogging, the flow rates in such a device are practically fixed to a maximum, except for slight variations through changes in hydrostatic level. This is undesirable because it is generally necessary to allow for flooding, especially at the beginning of a run, to insure thorough wetting of packing and also to take care of erratic or excessive flow rates without unduly upsetting the apportioning system.

Our invention is illustrated by the accompanying drawings, forming a part of this specification, in which:

Figure 1 is an elevational view of an embodiment of our invention in a still head for controlling the reflux ratio in fractionating columns with portions of the envelope broken away to show the interior construction;

Figure 2 is a partial side view of the same with portions of the envelope broken away to show the interior construction;

Figure 3 is a vertical section, at the position indicated by the line 3-3 in Figures 4 and 5, of

a portion of a multiple tube fractionating column embodying our invention;

Figure 4 is a horizontal section at the position indicated by the line 4—4 of Figure 3; and

Figure 5 is a horizontal section at the position indicated by the line 5—5 of Figure 3.

Referring now more particularly to Figures 1 and 2, the flow divider proper consists of an arc 6 carrying a winding of glass thread 7 and having a sphere 8 secured centrally on the top surface. The arc 6 is conveniently made from glass rod, the glass rod being formed into a curved segment of approximately circular curvature and drawn out at the ends to a conformation which will allow the reflux to flow easily off the ends. Other materials, such as ceramics or metallic rod, may also be used. The glass thread 7 wound on the arc 6 enhances the surface wetting properties of the arc. Wire may be used instead of glass thread, and etched grooves or embossed ridges also give good results. The sphere 8 can be made of any of the materials suitable for the arc 6. It provides a means to facilitate the uniform transfer of an incident stream over the whole surface of the arc 6.

Dimensions depend on the maximum rate of liquid reflux that the arc 6 is designed to carry. For liquid rates up to approximately 700 cc. per hour, a curved rod or arc of approximately 3 mm. cross-sectional diameter is serviceable. For higher rates, rod of somewhat larger diameter and with additional layers of fiber glass winding may be used. For much higher liquid rates (about 1500 cc. per hour) multiple arcs (not shown) made of two or more adjacent and parallel arcs with an over-all winding have been found serviceable. The diameter of the sphere 8 is approximately equal to the cross-sectional diameter of the arc 6 or in the case of multiple arcs (not shown) to the total width of the arcs taken together.

A supporting rod or tube 9 is joined to the side and center of the arc 6. The other end of the rod 9 extends through the center of a ground glass joint 10 and terminates in a lateral arm 11. The arm 11 is used for setting the angle of the arc for the desired ratio of liquid flow division or reflux ratio. The end of the arm 11 is drawn out to a tip 12 which may serve as a pointer on an empirically constructed scale (not shown), the reflux ratio obtained at a given position of the pointer 12 being determined by trial and marked on the scale.

The ground glass joint 13 connects the still head with the fractionating column below (not shown). Vapors arising from the fractionating column pass through the joint 13, up through the scarfed opening 14, and then into the tube 15 which leads to the condenser 16. The condenser 16 is provided with a water inlet 17, a water outlet 18, and a vapor outlet 19. The vapor outlet 19 may lead either to the atmosphere or to a vacuum pump. The condensed vapor or liquid reflux returns by gravity from the condenser 16 to the dropper 20 which is made of solid glass and is joined to the lowest part of the scarfed opening 14. The dropper 20 is fixed in a position directly above, and approximately 2 mm. distant from the sphere 8. The reflux liquid is fed by gravity from the dropper 20 to the sphere 8 from which it is spread evenly over the surface of the arc 6.

Depending on the position of the arc 6, which is controlled by the position of the arm 11, the liquid reflux can be made to enter the funnel

21 or the funnel 22, or can be divided between the funnels 21 and 22 in whatever ratio is desired. The volume of reflux liquid per unit of time entering the funnel 21, divided by the volume per unit of time entering the funnel 22, is the reflux ratio. For a given setting of the arc 6, the reflux ratio will be constant and substantially independent of the reflux rate or throughput.

The liquid in the funnel 21 is carried by gravity through the capillary tube 23 to a suitable receiver for collecting the product.

The liquid in the funnel 22 is carried by gravity to a dropper 24 which is joined to the bottom of the scarfed funnel outlet 25. The reflux liquid draining from the dropper 24 can be led by any convenient method through the joint 13 to the packing of the fractionating column below.

A glass envelop 26 serves as a housing for the apparatus and a mercury well 27 is arranged in this housing to accommodate a thermometer or thermocouple for registering the temperature of the vapors in the still head.

While certain of the elements of the embodiment of our invention just described have been specified as being made of glass, it should be noted that other materials, such as metals or ceramics, can be used as well.

Another application of our invention to fractionating columns is shown in Figures 3, 4, and 5. The apparatus illustrated constitutes a flow-dividing system for a multiple fractionating column, and is made up of several superimposed sections 28, 29, and 30 arranged in a housing 31.

Each section comprises a round perforated plate 32 having an annular flange 33 at its outer circumference. The flanges 33 are fitted with dowels 34 so that alignment between the sections can be maintained. The sections are supported in assembled form on brackets 35 attached to the housing 31. Openings 36 in the perforated plates 32 are arranged to provide free passage for rising vapors.

The perforated plate 32 of the bottom section 28 is designed to receive a plurality of hollow tubes 37 which extend vertically through and are joined below this plate 32 to spirally embossed rods 38 forming the actual packing elements of the fractionating column. The hollow tubes 37 serve as a feeding means for the rods 38, apertures 39 being arranged so that liquid will drain from the tubes 37 to the surface of the rods 38.

The spirally embossed rods 38, referred to above, are disclosed and claimed in our pending application for patent, Serial No. 424,476, filed December 26, 1941.

The present invention is concerned specifically with the liquid-distributing mechanisms as embodied in sections 29 and 30. However, the uniform distribution of liquid among the elements of a fractionating column is taken here only as an example. Our new type of liquid flow division and distribution apparatus could be applied with equal success to any apparatus in which the division of a liquid stream into smaller component streams is required.

The perforated plates 32 of sections 29 and 30, which are superimposed successively on section 28, are both fitted with a plurality of funnels 40 and 41, respectively, the upper end of the funnels being flush with the top surface of the plates and the lower end terminating in a scarfed opening. Attached to the lower tip of this scarfed opening is an adaption (arcs 42 and 43) of the flow divider previously described in connection with the apparatus for reflux control. As in that case, the

arcs 42 and 43, corresponding, respectively, with the funnels 40 and 41, are wound with glass thread 44, or otherwise provided with means to enhance their surface-wetting properties, and have a sphere 45 and 46, respectively, secured at the center of their top surface to distribute liquid evenly over the surface of the arc. The top of each sphere 45 and 46 is joined to the scarfed tip of the funnels 40 and 41 so that the spheres, and hence the arcs 42 and 43, are wetted by the liquid condensate flowing from the funnels. Spaced plates 47, forming an outline in plan around the openings 36 and the tubes 37 or the funnels 40 and 41 in the perforated plates 32, are secured to the plates 32 and extend upward to the full height of the flange 33 in each of the sections 28, 29, and 30. The plates 47 constitute a containing means which facilitates the passage of rising vapors through the openings 36. Also, the space between the plates 47 and the flanges 33 provides an insulating effect.

Each arc 42 and 43 is oriented and fixed in a symmetrical position so that it divides the stream issuing from the funnels 40 and 41 into two streams of equal volume.

In section 30 it is apparent that any arc 43 must carry twice the liquid volume of any arc 42 in the next lower section 29. Consequently, the arcs 43 and their attached spheres 46 are all of appropriately larger dimensions than the corresponding arcs 42 and spheres 45 in the next lower section 29. Likewise, for any other sections of flow-dividing elements that might be added, a similar progressive decrease in the dimensions of these elements in proceeding from a top section to a bottom section would be necessary. It is also apparent that section 29 must contain just twice as many flow-dividing elements as section 30.

The total number of sections of flow-dividing elements used will depend upon the total number of rods 38 comprising the packing elements in the fractionating tower. As illustrated the number of spirally embossed rods 38 is 128. More or less may be used as long as an even number is maintained.

For the purpose of illustrating the operation of this flow-dividing apparatus, assume that each rod 38 transfers on its surface, for operation at optimum efficiency, a liquid film of 100 cc. per hour. One arc 42 feeds the surfaces of one pair of rods 38. Therefore, there must be, in this particular column containing 128 packing rods 38, 64 arcs 42 and each arc 42 will carry 200 cc. per hour, or twice the liquid volume per hour carried by the rods 38. The funnels 40 transferring liquid condensate from section 29 to section 28 will also carry 200 cc. per hour and 64 of these funnels are required.

Similarly, one arc 43 and funnel 41 in section 30 will feed two funnels 40 and arcs 42 in section 29 so that the flow will again double and the arcs 43 and funnels 41 will carry 400 cc. per hour and only 32 of each are required. A section above section 30 would contain only 16 funnels, each with its attached sphere and arc, and each funnel would feed to its arc 800 cc. of liquid reflux per hour.

Proceeding from the lower to the upper sections, as the number of flow-dividing elements diminishes with concomitant increase in their individual capacities (cc. per hour), the need for a fine division of liquid becomes less exacting. At the point where one encounters high liquid transfer rates (1000 cc. or higher per hour), any of

the means described in our pending application for patent, Serial No. 424,476, filed December 26, 1941, or any of the conventional forms for distributing liquid reflux may be satisfactorily used.

A cycle in the apparatus proceeds as follows: Vapors rising from the fractionating column pass upward through the openings 36 in the perforated plates 32 and are taken off to a condenser (not shown). The return flow from the condenser is handled by some such means as mentioned in the preceding paragraph until the rate of flow decreases to the point where the flow-dividing means of this invention becomes necessary to effect proper distribution. Assuming that this point is reached in section 30, the reflux is delivered in equal amounts to the funnels 41. The funnels 41 feed the arcs 43 which divide the flow equally and transfer it to the funnels 40. The funnels 40 in turn feed the arcs 42, and the arcs 42 supply equal portions to the hollow tubes 37 which drain to the surface of the packing rods 38 of the fractionating column.

Our improved arc form flow divider has a number of distinct advantages: (1) it lends itself to throttling down the per-unit flow to an extremely low rate; (2) it provides a simple means for adjusting the rate of flow with considerable precision and thus permits the maintenance of accurate reflux ratio or a high degree of uniformity of rate for the respective units of a multiple system; (3) the respective units are positive and dependable in action, and (4) the "hold-up" per unit is very small, which is an important factor in column efficiency. The positive action of the units is made possible by the wetting or wick-like action provided by the glass thread winding which insures against stagnation or inactivity of any unit of the multiple system as sometimes happens with a system of weirs.

Having thus described our invention, we claim:

1. A device for effecting liquid flow division comprising a rod-like curved segment of approximately circular curvature, said curved segment being circular in cross section and positioned in a substantially vertical plane with its concave surface lowermost, means forming a spiral surface configuration throughout substantially the entire length of said curved segment constructed and arranged to enhance its surface wetability, and a spherical member centrally secured on the convex surface of curvature of said curved segment and positioned to receive the liquid to be divided, said spherical member having a diameter approximately equal to the cross-sectional diameter of said curved segment.

2. A device for effecting liquid flow division comprising a rod-like curved segment of approximately circular curvature, said curved segment being formed at its ends for surface discharge of a liquid stream and being circular in cross section and positioned in a substantially vertical plane with its concave surface lowermost, means forming a spiral surface configuration throughout substantially the entire length of said curved segment and constructed and arranged to enhance its surface wetability, and a spherical member centrally secured on the convex surface of curvature of said curved segment and positioned to receive the liquid to be divided, said spherical member having a diameter approximately equal to the cross-sectional diameter of said curved segment.

3. A device for effecting liquid flow division comprising a rod-like curved segment of approxi-

mately circular curvature, said curved segment being formed at its ends for surface discharge of a liquid stream and being circular in cross section and positioned in a substantially vertical plane with its concave surface lowermost, glass thread wound on said curved segment throughout substantially its entire length constructed and arranged to enhance its surface wettability, and a spherical member centrally secured on the convex surface of curvature of said curved segment and positioned to receive the liquid to be divided, said spherical member having a diameter approximately equal to the cross-sectional diameter of said curved segment.

4. A device for effecting liquid flow division comprising a plurality of rod-like curved segments of approximately circular curvature, said curved segments being formed at their ends for surface discharge of a liquid stream and being circular in cross section and positioned in a substantially vertical plane with their concave surfaces lowermost and being aligned parallel and adjacent, glass thread wound on said curved segments in an overall manner throughout substantially their entire length constructed and arranged to enhance wetting, and a spherical member centrally secured on the convex surface of curvature of said curved segments, said spherical member having a diameter approximately equal to the sum of the cross-sectional diameters of said curved segments and positioned to receive the liquid to be divided.

5. In a still head for fractionating columns and the like, means for returning condensate in a stream to said still head, a rod-like curved segment of approximately circular curvature, said curved segment being formed at its ends for surface discharge of a liquid stream, means forming a spiral surface configuration throughout substantially the entire length of said curved segment for enhancing its surface wettability, a spherical member centrally secured on the convex surface of curvature of said curved segment, said spherical member being of sufficient size for surface transfer to said curved segment of the stream of returning condensate and the diameter of said spherical member and the cross-sectional diameter of said curved segment being approximately equal, means for tiltably supporting said curved segment with its plane of curvature in a vertical position and the spherical member disposed upward in the line of incidence of said stream of returning condensate, said supporting means being adapted to allow the vertical position of the ends of said curved segment to be varied with respect to each other without removing said spherical member from the line of incidence of said stream of returning condensate, funneling means disposed under one end of said curved segment forming a conduit to the outside of said still head, and a second funneling means disposed under the other end of said curved segment for transferring liquid from said still head to a fractionating column.

6. In a still head for fractionating columns and the like, means for returning condensate in a stream to said still head, a rod-like curved segment of approximately circular curvature, said curved segment being formed at its ends for surface discharge of a liquid stream, glass thread wound on said curved segment throughout substantially its entire length, a spherical member centrally secured on the convex surface of curvature of said curved segment, said spherical member being of sufficient size for surface transfer to

said curved segment of the stream of returning condensate and the diameter of said spherical member and the cross-sectional diameter of said curved segment being approximately equal, means for tiltably supporting said curved segment with its plane of curvature in a vertical position and the spherical member disposed upward in the line of incidence of said stream of returning condensate, said supporting means being adapted to allow the vertical position of the ends of said curved segment to be varied with respect to each other without removing said spherical member from the line of incidence of said stream of returning condensate, funneling means disposed under one end of said curved segment forming a conduit to the outside of said still head, and a second funneling means disposed under the other end of said curved segment for transferring liquid from said still head to a fractionating column.

7. A flow dividing system for fractionating columns and the like comprising a housing, a plurality of superimposed flanged plates supported in said housing, said plates being disposed in spaced relation by the flanges thereon and having perforations to allow vapors to rise through said housing, an even number of funneling means fitted in each of said superimposed flanged plates, the number of said means progressively decreasing by one-half in each superimposed flanged plate, a spherical member attached in the line of liquid flow to each of said funneling means in the flanged plates above the lowest of said superimposed flanged plates, said spherical members being of sufficient size for surface transfer of the liquid flow from said funneling means, a rod-like curved segment of approximately circular curvature secured at the central point of its convex surface of curvature to each of said spherical members, said curved segments being formed at their ends for surface discharge of a liquid stream and the cross-sectional diameter of said curved segments being approximately equal to the diameter of said spherical members, and means forming a spiral surface configuration throughout substantially the entire length of each of said curved segments for enhancing their surface wettability, each set of funneling means, spherical members and curved segments being in vertical alignment, and each of said curved segments having its ends fixed in the same horizontal plane, disposed downwardly, and vertically aligned with funneling means in the flanged plate next below.

8. A flow dividing system for fractionating columns and the like comprising a housing, a plurality of superimposed flanged plates supported in said housing, said plates being disposed in spaced relation by the flanges thereon and having perforations to allow vapors to rise through said housing, an even number of funneling means fitted in each of said superimposed flanged plates, the number of said means progressively decreasing by one-half in each superimposed flanged plate, a spherical member attached in the line of liquid flow to each of said funneling means in the flanged plates above the lowest of said superimposed flanged plates, said spherical members being of sufficient size for surface transfer of the liquid flow from said funneling means, a rod-like curved segment of approximately circular curvature secured at the central point of its convex surface of curvature to each of said spherical members, said curved segments being formed at their ends for surface discharge of a liquid stream and the cross-sectional diameter of said curved

110

2,369,913

5

segments being approximately equal to the diameter of said spherical members, and glass thread wound on said curved segments throughout substantially their entire length, each set of funneling means, spherical members and curved segments being in vertical alignment, and each of said curved segments having its ends fixed in the

same horizontal plane, disposed downwardly, and vertically aligned with funneling means in the flanged plate next below.

5 *Executrix of the Last Will and Testament of Samuel Palkin, Inventor, Deceased.*

ROSE PALKIN,

STANLEY A. HALL.