April 27, 1943. R. E. BURK ET AL 2,317,952

FRACTIONAL DISTILLATION

Filed Nov. 1, 1940

FIG. 2

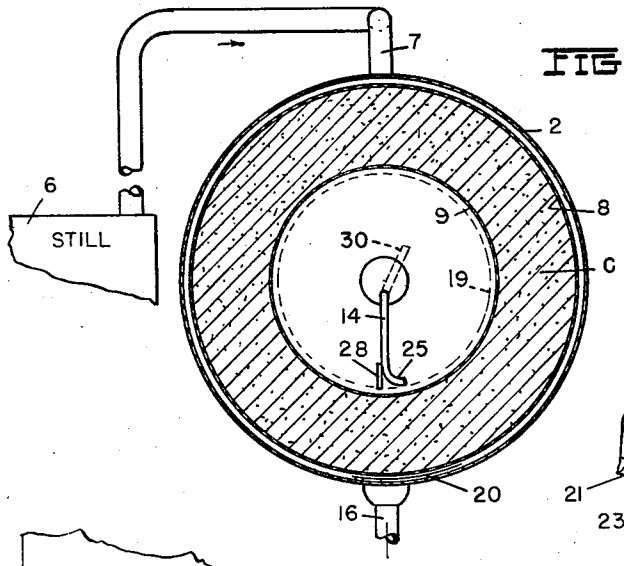


FIG. 3

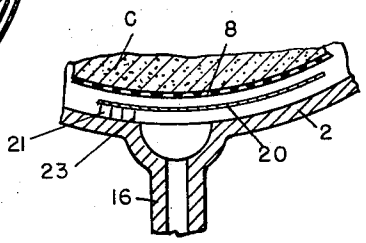


FIG. 1

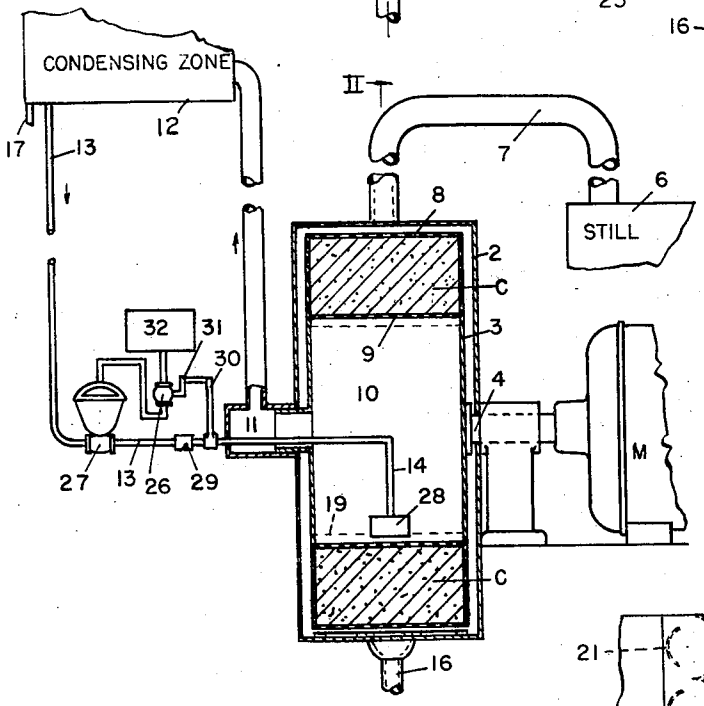
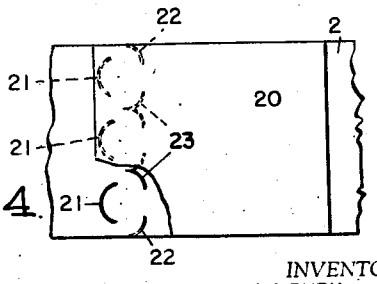


FIG. 4



INVENTORS
ROBERT E. BURK
and
EINAR KROPP

BY Oberlin, Limbach & Day
ATTORNEYS.

Patented Apr. 27, 1943

2,317,952

UNITED STATES PATENT OFFICE

2,317,952

FRACTIONAL DISTILLATION

Robert E. Burk, Cleveland Heights, and Einar Kropp, Cleveland, Ohio, assignors to The Standard Oil Company, Cleveland, Ohio, a corporation of Ohio

Application November 1, 1940, Serial No. 363,854

18 Claims. (Cl. 261—83)

In fractional distillation, it has been generally customary to employ bubble plate columns or packed towers, and where considerable accuracy of separation and high through-put have been desired, the size and the space requirements for such columns run into very large dimensions, which are objectionable, and in some cases prohibitive. In contrast, to attain great through-put with very small size apparatus, provision is made in the patent application of Arthur G. McKee, Ser. No. 217,086, since matured into U. S. Patent 2,259,762, dated Oct. 21, 1941, to carry on the fractionating in a mechanically driven apparatus providing accentuated contact between the vapors and condensing liquid. The present invention is an improvement in the latter type of apparatus; and makes possible the attainment of very high accuracy of separation, together with sustained high through-put and control.

To the accomplishment of the foregoing and related ends, the invention, then, comprises the features hereinafter fully described, and particularly pointed out in the claims, the following description and the annexed drawing setting forth in detail certain illustrative embodiments of the invention, these being indicative however, of but a few of the various ways in which the principle of the invention may be employed.

In said annexed drawing:

Fig. 1 is an axial sectional view of apparatus in accordance with the invention; Fig. 2 is a transverse section taken on a plane substantially indicated by line II, Fig. 1; Fig. 3 is an enlarged detail of the lower portion of Fig. 2; and Fig. 4 is a plan view of the structure shown in Fig. 3.

The apparatus involves a housing 2 within which is rotatably carried an annular contact body C for vapor and liquid, suitable carrier means 3 therefor being mounted with a shaft 4 and driven by suitable means, for instance a motor M. Vapors from a distillation zone or still 6 are introduced into the housing 2 by an inlet connection, whence they pass through the pervious outer periphery 8 and through the contact mass C and thence through the inner pervious periphery 9 to the central space within the annulus, and on through an outlet connection 11 to a condensing zoe 12. From the latter, reflux liquid may be returned by a connection 13 and inlet 14 to the inner periphery of the annular body C. Liquid is taken off from the housing 2 by an outlet 16. and condensate product from the condensing zone by a discharge connection 17.

In fractionating in apparatus involving rotary means, the sorting out of molecules occurs not only at the liquid and vapor inter-faces where liquid films cover the various surfaces of the pervious body, but if a controlled layer or ring of liquid be present on the inner periphery of the rotating body a particularly increased field of action is provided. The reason for this latter may be understood when it is noticed that whereas in a bubble tower there are small fringes of bubbles passing from under each cap out through the layer of liquid on the tray, here an enormous number of fine bubbles can escape generally from the multitudinous openings in the inner periphery of the pervious body and pass through a layer of liquid if present, thereby affording small bubbles and liquid contact on a multiplied and accelerated scale. It is particularly important that any such layer or ring of liquid be constant and of efficient depth or thickness. If varying from nothing to a surplus, poor fractionation results, and if there is variation generally, proper fractionation is not obtained. A feature of the present invention is the provision of an inner layer of liquid 19, such that constancy of presence and uniformity of depth are maintained and uniformity of return of reflux liquid through the mass. In some cases we accomplish this by control of the angular velocity of the rotating body, size of interstitial passages, rate of supply of vapors, etc., or with particular convenience certain or all of these factors may be made constant and control may be exercised by the amount of reflux feed. And the invention contemplates control in general, by any of the various possible means. Preferably, control is made automatic. Thus for instance, a means responsive to variations in depth in a layer of liquid on the inner periphery of the rotating body may be arranged to control the feed-valve 27 of a reflux line. The layer-responsive means may be mounted in any convenient manner, but for instance may take the form of a member 28 of extent to cause resistance to liquid, secured on the reflux feed conduit 14 and set at such a height relative to the inner periphery of the rotatable body that when the liquid layer is of predetermined height it tends to drag the member 28 and the tube 14 in its direction. By mounting the tube for slight turning movement on its own axis, and with a swivel-permitting connection 29 to the main pipe 13, the tube 14 can have movement through a sufficient arc to actuate a pilot control device or valve 26 to regulate a diaphragm valve 27 on the reflux line. Thus, an arm connection 30 gives sufficient movement to the valve lever 31, and this acts to supply pressure fluid, pneumatic or hydraulic, from tank

32 to set valve 27 which governs the supply of reflux. When the liquid level 19 declines and does not drag the responsive member 28 the latter swings back by its weight to normal position, closing valve 26, and by having a diaphragm chamber on valve 27 of vented character to allow leakage and release of pressure fluid, when the line-pressure from supply 32 is cut off, the valve 27 opens and permits flow of reflux. The action of member 28 should be subjected to constant torque conditions at all times in order that the same deflections may be had for the same reflux level.

Ordinary arrangements for returning reflux result in some splash and entrainment. But, by delivering the reflux tangentially in the direction of rotation as provided by the arrangement of the reflux discharge 25 splash and its resulting entrainment is eliminated.

With advantage, the discharge outlet may be constructed as in accordance with the patent application of Everett C. Hughes, Ser. No. 363,863, since matured into U. S. Patent No. 2,298,294 dated Oct. 13, 1942, viz. over the opening of the outlet 16 there is positioned a spaced cover baffle 20, which extends circumferentially beyond the opening in both directions, and particularly in the direction of rotation. Under this, at the leading edge, are baffles 21, 22 and 23, in staggered position.

It has been very difficult to attain suitable uniformity of interstitial spaces in materials heretofore applied for making up the rotary contact body. Even if wholly uniform materials such as wire mesh were employed, unevenness developed in placement of layer upon layer in stacking or winding. Furthermore, where corrosion of filling occurs, there is still further interference with uniformity and accurate fractionating. We prefer to make up the rotatable body C of solid particles of uniform grain, and of non-corrodible character. Thus, silicon carbide or "carborundum," porcelain shot, saddles, rings, etc., are uniformly gradable and of accurate sizes within the range suitable to provide spacing for fractionating substances suitably amenable to fractional distillation. The rotatable carrier 3 is provided with such filling before its assembly within the housing 2, and a further result and advantage of the use of such kind of filling is that where substances are being distilled which tend to form a residue or coating upon the surfaces of the filling, cleaning may be accomplished as desired, without damage to the filling, by suitable solvents or by combustion sufficient to burn the coating off, the metal parts being such as to permit.

In operation, vapors to be fractionated are supplied to the housing 2 from the vapor supply connection 7, and pass into the rotary pervious annular body C from the outer periphery. The pervious rotor C being in suitable rotation by its driving means, reflux liquid is simultaneously supplied from the line 13. Discharging in the direction of rotation tangentially, the liquid quietly builds up a layer on the annular rotating surface and portions progressively pass outwardly under the centrifugal action, countercurrently meeting vapors, the uncondensing portions of which proceed through the layer 19 of liquid on the inner periphery, the contact there being by multitudinous fine bubbles as the vapors emerge from the small openings at the surface of the inner periphery. A sufficient pressure differential being provided, by raised pressure at the feed side or by reduced pressure at the condensing zone, or both, as preferred in any particular instance, the vapors proceed from the central space 10 to the outlet 11 and to the condensing zone 12. Reflux therefrom, as desired, thence feeds back through the line 13 to the inner periphery of the rotor. As indicated, the depth of the liquid layer 19 on the inner periphery of the rotor may be controlled as desired by suitable regulation of the driving rate of the rotor or by regulation of the rate of vapor or reflux feed, or particularly desirably the depth may be controlled by the depth-responsive member 28 being subjected to drag in the direction of rotation when the liquid level reaches a depth such as to engage it. With the movement of the responsive member 28 to a slight extent in the direction of rotation of the liquid layer engaging it, the angular arm 30 is swung with the pipe and in turn, through its direct cross connection to the valve lever 31 swings it sufficiently to operate the valve 26 to apply fluid pressure from the control supply 32 to the diaphragm valve 27, and thereby correspondingly close the latter and check the flow of reflux through the pipe 13 to the rotor. Conversely, with cessation of drag movement of the responsive member 28, its weight returns it to normal position, thereby swinging the arm 30 and valve stem 31 back to shut off the pressure operating from and the valve 27 and allow it to then open. Regulatory compensating valve action in such manner correspondingly proceeds and maintains a liquid layer 19 at a predetermined thickness, with resultant uniformity of exposure for bubbling vapors and uniformity of distribution into and through the pervious rotor 6. Liquid discharging from the rotor proceeds from the housing to discharge outlet 16. The lower boiling final product of the fractionation is taken off from the condensing zone by discharge connection 17.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims, or the equivalent of such, be employed.

We therefore particularly point out and distinctly claim as our invention:

1. In apparatus of the character described, a rotatable fluid-pervious annular mass, means for rotating same, a connection for passing vapors from the space within said annular mass to a condensing zone, and a connection entering coaxially for returning reflux liquid and being bent to deliver the liquid tangentially on the inner periphery of said annular mass in the direction of its rotation.

2. In apparatus of the character described, an annular fluid-pervious mass of carborundum, means for rotating the same, a connection for passing vapors from the space within said annular mass to a condensing zone, and a connection entering coaxially for returning reflux liquid from a condensing zone and being bent to deliver the liquid tangentially to the inner periphery of said annular mass in the direction of rotation.

3. In apparatus of the character described, an annular contact body for vapor and liquid, a rotatable carrier therefor pervious to fluids at its inner and outer peripheries, an external closed housing, means for introducing vapors into the housing, a condensing zone outside of said housing, an outlet for vapors from the central space inside the annular body to said condensing zone, means for returning liquid from said condensing

zone on the inner periphery of the annular body and valve means controlled by the depth of such liquid for maintaining its depth as a substantially constant annular layer thereon.

4. In apparatus of the character described, an annular mass of carborundum grains, a rotatable carrier therefor pervious to fluids at its inner and outer peripheries, a connection for discharging liquid on the inner periphery, valve means controlled by the depth of such liquid for maintaining its depth as a substantially constant annular layer thereon an external housing, means for introducing vapors into the housing, an outlet for vapors from the central space inside the annular mass to a condensing zone, and an outlet for liquid from the housing.

5. In apparatus of the character described, an annular mass of small porcelain bodies, a rotatable carrier therefor pervious to fluids at its inner and outer peripheries, a connection for discharging liquid on the inner periphery, valve means controlled by the depth of such liquid for maintaining its depth as a substantially constant annular layer thereon an external housing, means for introducing vapors into the housing, an outlet for vapors from the central space inside the annular mass to a condensing zone, and an outlet for liquid from the housing.

6. In apparatus of the character described, an annular mass of small non-corrodible bodies, a rotatable carrier therefor pervious to fluids at its inner and outer peripheries, a connection for discharging liquid on the inner periphery, valve means controlled by the depth of such liquid for maintaining its depth as a substantially constant annular layer thereon an external housing, means for introducing vapors into the housing, an outlet for vapors from the central space inside the annular mass to a condensing zone, and an outlet for liquid from the housing.

7. In a process of separating substances having different boiling temperatures by moving liquid in film form by centrifugal force over irregular surfaces travelling in an annular path and forcing vapors of higher temperature than the liquid in countercurrent flow thereto through the interstices between the films on the irregular surfaces, establishing a rotating annular layer of reflux liquid on the inner periphery of the annular traveling irregular surfaces, and maintaining a desired depth of the annular layer of liquid by means responsive thereto governing the amount of reflux fed to such annular layer of liquid.

8. In a process of separating substances having different boiling temperatures by moving liquid in film form by centrifugal force over irregular surfaces travelling in an annular path and forcing vapors of higher temperature than the liquid in countercurrent flow thereto through the interstices between the films on the irregular surfaces, inhibiting entrainment of splashed liquid in the vapor by supplying the liquid to the inner periphery of the annular traveling irregular surfaces in the direction of rotation.

9. In apparatus having an annular contact body for vapor and liquid and a rotatable carrier therefore pervious to fluids at its inner and outer peripheries in an external housing with means for introducing vapors into the housing and an outlet for vapors from the central space inside the annular body to a condensing zone, means for providing a layer of liquid on the inner periphery of the annular body, said means including a connection for feeding reflux liquid from a condenser zone to the inner periphery of the annular body, a valve therefor, means movable by the drag of the liquid layer at a predetermined depth, and means actuated by the latter means for controlling said valve.

10. In apparatus having an annular contact body for vapor and liquid and a rotatable carrier therefor pervious to fluids at its inner and outer peripheries in an external housing with means for introducing vapors into the housing and an outlet for vapors from the central space inside the annular body to a condensing zone, means for providing a layer of liquid on the inner periphery of the annular body, said means including a connection for feeding reflux liquid from a condenser zone to the inner periphery of the annular body, and means operated by the layer of liquid on obtaining a predetermined depth for controlling the feed of reflux.

11. In apparatus having an annular contact body for vapor and liquid and a rotatable carrier therefor pervious to fluids at its inner and outer peripheries in an external housing with means for introducing vapors into the housing and an outlet for vapors from the central space inside the annular body to a condensing zone, means for providing a layer of liquid on the inner periphery of the annular body, said means including a connection for feeding reflux liquid from a condenser zone to the inner periphery of the annular body, and means operated by variation in depth of such liquid for controlling the reflux feed.

12. In apparatus of the character described, an annular mass of carborundum grains, a rotatable carrier therefor pervious to fluids at its inner periphery, a connection for passing vapors from the central space within said annular mass to a condensing zone, a connection for returning reflux liquid from a condensing zone, an oscillably mounted responsive member arranged in the path of liquid on the inner periphery of said annular mass, a valve controlled by said responsive member, and a valve on the reflux return connection controlled by said first-mentioned valve.

13. In apparatus of the character described, an annular mass of small non-corrodible bodies, a rotatable carrier therefor pervious to fluids at its inner periphery, a connection for passing vapors from the space within said annular mass to a condensing zone, a connection for returning reflux liquid from a condensing zone to the inner periphery of the annular mass, and means governed by the height of reflux layer on the inner periphery of said mass for controlling the reflux feed from the reflux connection to such liquid layer.

14. In apparatus of the character described, a rotatable fluid-pervious annular mass, means for rotating same, a connection for passing vapors from the space within said annular mass to a condensing zone, and liquid-condensate feed means from a condensing zone governed by the height of liquid on the inner periphery of said annular mass for supplying liquid thereto.

15. In apparatus of the character described, a rotatable fluid-pervious annular mass, means for rotating same, a connection for passing vapors from the space within said annular mass to a condensing zone, and means for maintaining a controlled height of liquid on the inner periphery of said annular mass, including a connection from a condensing zone and a control valve therefor and a pilot valve for moving said control

valve and actuated by the height of liquid in such layer.

16. In apparatus of the character described, a rotatable fluid-pervious annular mass, means for rotating same, a connection for passing vapors from the space within said annular mass to a condensing zone, and means for maintaining a controlled height of liquid on the inner periphery of said annular mass, including a connection from a condensing zone and valve means governed by the height of liquid in such layer for controlling said valve means.

17. In apparatus having a rotatable fluid-pervious annular mass with means for rotating the same and a connection for passing vapors to a condensing zone from within said annular mass, a pipe for returning reflux liquid from said condensing zone, a swiveled connection from said pipe leading to discharge upon the inner periphery of said annular mass and normally hanging in pendant position, a liquid-pressed surface on the back of such pendant connection actuated by a predetermined height of liquid layer on the inner periphery of said mass to swing the connection in the direction of rotation of the liquid, a lever from said connection and a fluid-pressure valve operated thereby, a diaphragm valve in said reflux pipe, and means whereby said diaphragm valve is controlled by said fluid-pressure valve for controlling the amount of reflux fed through said connection.

18. In apparatus having a rotatable fluid-pervious annular mass with means for rotating the same and a condensing zone receiving vapors from within said annular mass, a pipe for returning reflux liquid from said condensing zone, a swiveled connection from said pipe leading to discharge upon the inner periphery of said annular mass and normally hanging in pendant position, a liquid-pressed surface on the back of such pendant connection actuated by a predetermined height of liquid layer on the inner periphery of said mass to swing the connection in the direction of rotation of the liquid, and a valve on said reflux pipe operated by movement of said swinging connection for controlling the amount of reflux fed to the rotatable annular mass.

ROBERT E. BURK.
EINAR KROPP.