Jan. 9, 1951 H. S. MARTIN 2,537,942

DISTILLING HEAD

Filed July 16, 1947

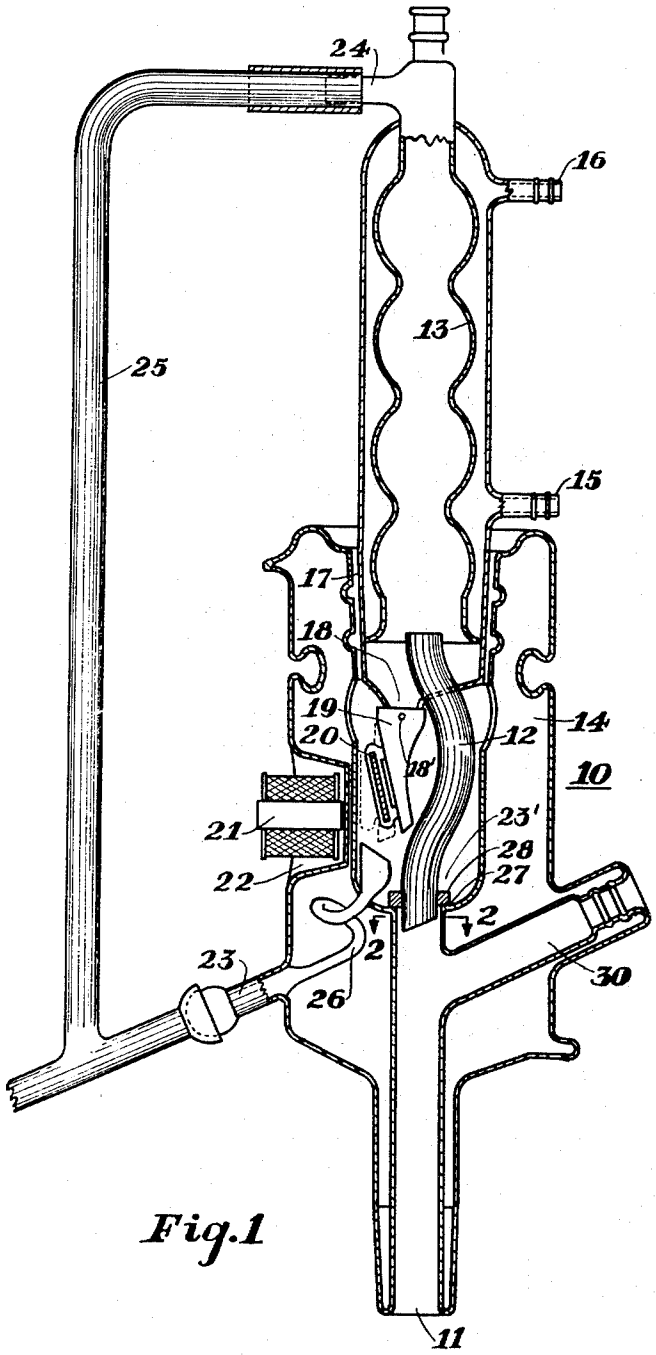


Fig. 1

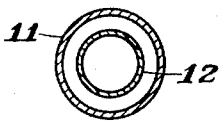


Fig. 2

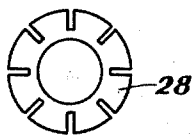


Fig. 3

INVENTOR.
Howard S. Martin
BY
Edward H. Lang
ATTORNEY

Patented Jan. 9, 1951

2,537,942

UNITED STATES PATENT OFFICE

2,537,942

DISTILLING HEAD

Howard S. Martin, Glenview, Ill., assignor to The Pure Oil Company, Chicago, Ill., a corporation of Ohio

Application July 16, 1947, Serial No. 761,374

5 Claims. (Cl. 202—161)

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This invention relates to an improvement in distilling apparatus for use in conducting laboratory and precise analytical distillations.

In United States Patent 2,251,185, issued July 29, 1941, to Carter and Johnson, a reflux control apparatus for use in a fractional distilling column is disclosed. As described in the patent, the apparatus has several distinct disadvantages, the principal one of which is the vapors from the distilling column enter the product section surrounding the swinging product guiding funnel for receiving product and are partially condensed and returned to the fractionating column without passing through the funnel, thereby rendering it impossible to regulate accurately the amount of reflux liquid returned to the distilling column. Another disadvantage of the apparatus is that it is constructed in a manner which makes it impossible to gain direct access to the funnel. When it is considered that the still in use is subject to frequent accidents and breakage in that particular portion of the device, this constitutes a serious obstacle to its efficient and economical use. Likewise, the distillation of a material containing gum forming compounds invites the formation of gum on the funnel supports and the funnel itself, thereby interfering with its free operation and necessitating disassembly in order to correct the operating difficulties.

Accordingly, it is an object of the instant invention to provide a distilling apparatus which is unitary in form, but can be disassembled at a plane approximately corresponding to the level at which the guiding funnel is suspended.

A further object of the invention is to provide a distilling apparatus which serves to divide positively the overhead condensate to deliver it either externally as product, or internally as reflux back to the distilling column for revaporization.

It is a further object of the invention to make it possible to adjust the heat content of the condensate being returned to the column as reflux by allowing it to flow counter-current and in indirect heat transfer relationship with the hot vapors rising through the product directing zone of the apparatus.

Other objects and advantages of the invention will in part be obvious and in part appear hereinafter.

The invention, accordingly, comprises a precision distillation apparatus which includes the features of construction, combinations of elements and arrangements of parts hereinafter described, and embodied in the distilling apparatus illustrated in the accompanying drawings.

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In the accomplishment of the objects of this invention, the structure is made to include means for delivering product externally or internally as reflux, the division of product thereby making possible a time controllable reflux : product ratio enhanced in accuracy by the structural features of the apparatus which include a swinging condensate guiding funnel, a condensate guiding funnel zone practically free of condensable vapors, conduit constituting an upper portion of the distilling column which, by virtue of its central location, is shielded from ambient temperatures by a vacuum jacket and acts as a normalizing or steadying influence in the temperature registration of the distilling vapors. With this structure, revaporization of reflux is easily accomplished in the column and, because flooding is thus minimized, a very substantial increase in the overall capacity of the column without loss of fractionating ability results.

Referring to the drawings which show one embodiment of the invention;

Figure 1 is a longitudinal sectional elevation of the apparatus;

Figure 2 is a horizontal sectional view of the apparatus taken along the line 2—2;

Figure 3 is a plan view of a ring seal forming a part of the apparatus.

In the drawing, there is shown a jacketed condenser and distilling head 10 having a vapor inlet and conduit 11, vapor conduit 12 through the apparatus to conduct vapors into condenser 13, shown with a corrugated interior condensing surface.

Conduits 11 and 12 lead from a distilling column, or source of vapors, and are enclosed by vacuum jacket 14 which consists of a properly shaped envelope surrounding vapor inlet 11 and conduit 12, and extends upwardly an operable distance on condenser 13. For efficient heat conservation, and to provide for regulation of temperature to a stable level, it will generally be found advisable to have the interior of the jacket silvered. Vapor conduit 12 is an integral part of condenser 13 being fused in the lower end thereof and removable therewith. Condenser 13 is supplied with cooling liquid via inlet and outlet 15 and 16, respectively.

Condenser 13 is fitted to the jacket 14 by means of an accurately made ground glass joint 17 which is preferably sealed with a good vacuum cement when the apparatus is used. Pivotally suspended from the lower end at product outlet 18 of the condenser by means of pin 18' is the product guiding funnel 19 which is equipped with a mag-

netic arm 20 in order to provide for movement thereof from the exterior. Electromagnet 21 placed in recess 22 in envelope 14 is effective when activated to move funnel 19 into the position shown in dotted lines. Product line 23, having an enlarged inlet end, leads from the enclosed product collecting zone 23' of the head through jacket 14 to the exterior. Vapor outlet 24 on condenser 13 provides for pressure equalization in the condenser with that in the product section and line by means of conduit 25, connected, for example, at 24 by a hose connection and at 23 by a cemented ground glass connection. Product forming in the condenser is discharged from product outlet 18 into funnel 19 which is pivoted thereon. Product can be returned to the evaporating zone or delivered to the product line 23 via conduit 26. The enlarged inlet end of product line 23 is placed in a position to receive product when the magnet 21 is activated to draw funnel 19 into product delivery position.

Return of condensate to the vaporizing zone is accomplished via a restricted opening at shoulder 27 which preferably is formed by a ring-shaped member 28, having slots therein and resting on the shoulder 27 where it overlies the annulus formed by conduits 11 and 12. A detail of the ring 28 is shown in Figure 3, where it appears as a ring having a series of regularly spaced radial slots spaced along its periphery. Since the purpose of ring 28 is to restrict the area of the annulus for return of product, the ring can take a variety of forms in which serrations would define the openings, the limiting form being one in which a single small orifice would constitute the opening. It is also possible to construct conduits 11 and 12 to a close tolerance such that the annulus alone would restrict flow sufficiently, but that expedient is structurally impracticable.

In the side of the apparatus at a convenient level in the vapor conduit 11, provision is made for insertion of a thermometer by providing well 30 therefor.

The operation of the condenser is readily understood simply by tracing the drawing in the manner described. The vapors leave a still or vaporizing zone, pass up through conduit 11 and follow the path of least resistance which takes them through conduit 12, up into condenser 13 where condensation takes place on the side walls. Condensate drains down and collects toward the depressed outlet 18, where it is discharged into collecting and guiding funnel 19. When it is desired to return condensate to the vaporization zone for reboiling, the funnel is allowed to depend in the position shown and thus, liquid will collect around ring 28 in the product collecting zone 23'. Liquid will quickly seal the slots in the ring and after it collects to a slight level within the collection zone, it will possess sufficient head to pass through the slots and back into the vaporization zone. When product is being taken out through line 23, a condition of dynamic equilibrium exists at 28, for just enough liquid will be held on the ring to seal the openings and vapors will pass through wide open conduit 12 to condenser 13. When it is desired to collect product at a given rate, the electromagnet 21 is activated to attract magnetic arm 20 in the funnel causing it to be drawn into the position shown in dotted lines, whereby its delivery spout is aligned with the enlarged receiving end of product line 23.

In order that product may flow unhindered

out of the condenser, equalization of the pressure in the condenser and that prevailing in the product line is accomplished by connecting the two zones. It can be seen that in operation, vapors are conducted directly from the vaporization zone into the condenser and do not come into direct contact and only into indirect heat transferring relationship with the product being returned to the funnel. This provides a distinct advantage in operation in that precise control over the condensate is maintained. Likewise, by proper choice of the length of the condenser 13 and length of vacuum jacket 14, it is possible to pass vapors directly into the condenser without creating any appreciable degree of superheat.

What is claimed is:

1. A distilling head comprising, a first insulated vapor passageway for connection to a still as a source of vapors, a vacuum insulated jacket extending upward from the upper end of said vapor passageway to define an insulated condensate receiving section around the upper end of said vapor passageway, a jacketed condenser for connection to the upper end of said condensate receiving section, said condenser having an open bottom end to deliver condensate to said condensate receiving section, a second vapor conduit connecting said first vapor conduit with the lower section of said condenser to form a vapor passageway through said condensate receiving section and into the lower end of said condenser, a pivoted funnel mounted within said condensate receiving section immediately below the open end of the said condenser, a conduit for withdrawing condensate received from the funnel, an annular opening surrounding the lower end of said second vapor passageway and defined by the second vapor passageway and the condensate receiving section, the opening being covered by a serrated ring, the serrations of which restrict the free area of said annulus to allow liquid to return from said receiving section to said first mentioned passageway without allowing vapors from said first mentioned vapor passageway to enter said condensate receiving section when condensate is present in said receiving section.

2. A distilling head in accordance with claim 1 which includes two sections, a condenser section and a vapor conduit and vacuum jacketed product section, the one fitting the other closely to permit operation thereof, the condenser section being encased within the vacuum jacketed product section to bring the vacuum jacket to a level corresponding approximately to the height of the exit of vapors from the vapor line and the point of entry thereof into the condenser.

3. An apparatus in accordance with claim 1 wherein the vapor inlet conduit and condenser are arranged vertically in series, the vapor inlet to the condenser being at substantially the lowest cool level thereof, the whole being surrounded by an evacuated envelope extending from a point substantially below the vapor inlet to a point just above the lowest point of the cooling surface of the condenser.

4. A distilling head comprising, a first insulated vapor passageway for connection to a still, a condensate receiving section around the upper end of said passageway, a jacketed condenser attached to the upper end of said condensate receiving section, said condenser having an open bottom end to deliver condensate to said condensate receiving section, a second vapor passageway of lesser cross-sectional area than said

first passageway leading from the first vapor passageway through said condensate receiving section and into the lower end of said condenser, a pivoted funnel mounted within said condensate receiving section immediately below the open bottom end of said condenser, a product conduit for withdrawing condensate received from said funnel and an annular passageway from said condensate receiving section to said first mentioned vapor passageway, said annular passageway being defined by overlapping of the ends of said first and second vapor passageways, a ring restricting said annular passageway, the restriction in area being sufficient to retain some liquid to form a seal in the annular passageway so that vapors from said first-mentioned vapor passageway enter said second vapor passageway without entering the condensate receiving section when condensate is present in said receiving section.

5. A distilling head comprising, a first insulated vapor passageway for connection to a still, a condensate receiving section around the upper end of said passageway, a jacketed condenser attached to the upper end of said condensate receiving section, said condenser having an open bottom end to deliver condensate to said condensate receiving section, a second vapor passageway of lesser cross-sectional area than said first vapor passageway leading from the first vapor passageway through said condensate receiving section and into the lower end of said condenser,

a pivoted funnel mounted within said condensate receiving section immediately below the open bottom end of said condenser, a product conduit for withdrawing condensate received from said funnel and an annular condensate return from said condensate receiving section defined by the walls of said first and second vapor passageways, the annular return being restricted in area by close dimensioning of the said respective vapor passageways, said restriction in area being sufficient to retain some liquid to form a seal so that vapors from said first mentioned vapor passageway enter said second vapor passageway without entering the condensate receiving section when condensate is present in said receiving section.

HOWARD S. MARTIN.

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2,251,185	Carter et al.	July 1941

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