

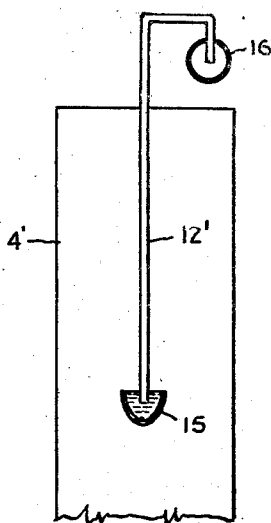
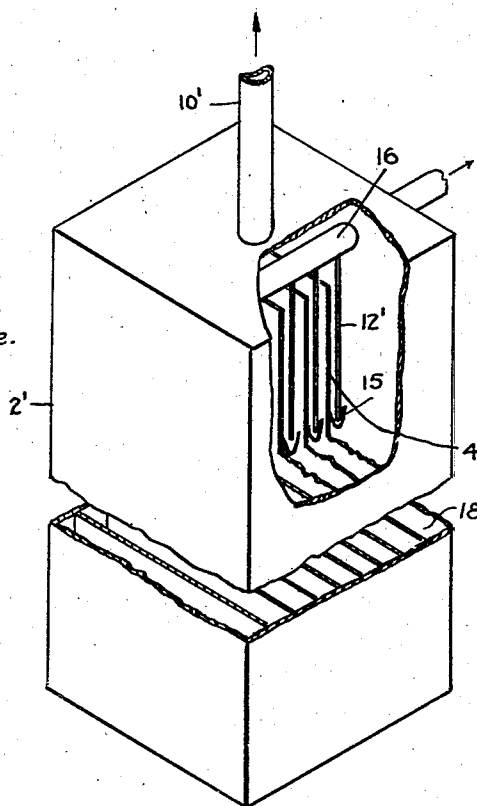
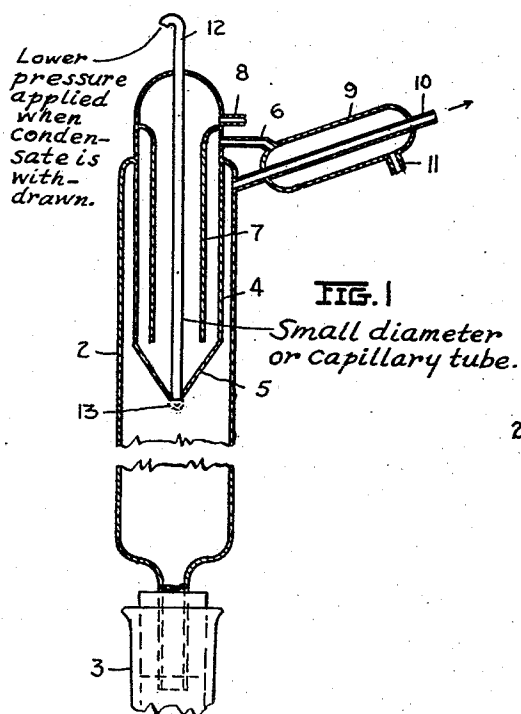
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LABORATORY FRACTIONATING COLUMN HEAD.

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LABORATORY FRACTIONATING COLUMN HEAD

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In precision fractional distillation, particularly such as is involved in separation of mixtures of closely boiling constituents such as for determination of compositions, as notably for instance in the case of hydrocarbon mixtures etc., it is of great importance that the still head should be designed so that a minimum of the liquid being distilled is held in the still head. This has been lacking to such extent as desired, with customary designs of equipment. In accordance with the present invention, the amount of liquid held in the still head may be reduced to terms of a small drop in small laboratory apparatus, and proportionally in larger size apparatus; and furthermore, fractionation may be applied to a particularly small sample of material. Other objects and advantages will appear from the following description.

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 a side elevational view showing an embodiment of the invention, partly broken; Fig. 2 is a perspective, partly broken view of a vertical plate type of apparatus involving the invention; and Fig. 3 is a fragmentary elevational view of a detail.

In general, the surface upon which reflux condensate forms in the upper part of the fractionating apparatus is arranged so as to provide condensate in the form of a drop on the end opening of a small diameter or capillary take-off tube, such that material can be withdrawn from such drop as required, for instance by applying a vacuum. The apparatus may be made of glass, or other material as desired. For non-corrosive liquids and gases, metals such as copper, iron, steel, brass, zinc, etc. may be used. With liquids and gases which might cause corrosion, stainless steel, high chromium steel, nickel, Monel, etc. may be employed. Usually, glass is desired for the small laboratory columns, and as shown in Fig. 1, such an apparatus may involve a tubular member 2 suitably mounted on a distilling flask or the like 3, and having in its upper end a condensing surface of general cylindrical form 4, drawn in, as by rounding or tapering at its lower end 5, and having connections 6 and 8 for cooling fluid, and an

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internal tubular baffle wall 7 around the lower end of which the fluid may flow. A jacket 9 on the vent outlet 10 from the head 2 may also receive the cooling fluid and have a connection 11. Extending through the cylindrical condensing member 4 is a small diameter or capillary tube 12, its lower end opening out from the end 5 of the condensing surface such that the drainage from the condensing surface runs down to form a drop 13 which may be maintained at the lower end of the tube 12 by the surface tension of the liquid condensate. The other end of the tube 12 may be connected e. g. through a stopcock for application of such lower pressure as may be desired in order to draw off liquid from the drop. A vacuum for instance may be applied for such purpose, or, operation may be carried on at atmospheric pressure and a syphon may be used to remove the liquid.

The operation of the device will be understood from the foregoing. The liquid to be fractionated is suitably vaporized, as in the flask 3, and the vapors rising up through the tubular head 2 encounter the condensing surface 4, and condensate thereon drains down to establish a drop 13 on the end of the take-off tube 12. Vacuum is applied therethrough as desired, and draw-off from the standing drop of the liquid may be had into a receiver in the vacuum line. The liquid hold-up in the device being in drop dimensions, it is to be seen that the apparatus is particularly responsive to the components being fractionated, and great accuracy results.

In apparatus having its condensing surface in a form other than cylindrical, as for instance vertical parallel plates, as shown in Fig. 2 the small diameter or capillary tubes 12' may be applied in such numbers as desired, with the lower end of each tube provided with a small collector-cup 15 on the vertical plate condensing surface 4', such that in effect again a drop of condensate liquid is supplied to cover the outlet end of the capillary tube. The capillary tubes may be connected to a manifold 16 leading to a source of vacuum or lowered pressure to permit draw-off of liquid as desired into a receiver in the vacuum line. As many vertical surface plates may be provided in the fractionator 2' as desired, and an outlet vent 10' is provided at the top.

In the operation of the apparatus shown in Figs. 2 and 3 the vapor from the still ascends through the passageways 18 between the vertical condensing surfaces 4'. The vapor which condenses on one of the surfaces 4', as will be seen more particularly from a consideration of Fig. 3,

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flows down the condensing surface due to the action of gravity. A portion of this condensate which flows down the surface immediately above the collector-cup 15 is collected in the cup which is mounted on the condensing surface. After the collector-cup 15 is full, additional condensate collecting in it flows over the side of the cup and returns to the still. The small diameter or capillary tubes 12' are so spaced that the lower opening in the tube projects into the collector-cup 15, so that the opening is covered by the condensate collected in the cup. When it is desired to withdraw a portion of the liquid, the pressure in the manifold 16 may be lowered, whereupon the condensate collected in the cup 15 will rise through the tube 12' and flow into the manifold 16 and may be withdrawn therefrom.

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. A fractionating column head, comprising a chamber, a condenser including a generally cylindrical condensing surface projecting into such chamber at its upper end, said condensing surface being tapered at its lower end, a capillary tube opening at the lower end of the condensing surface and extending out of said condenser to connect to a source of lowered pressure, cooling fluid connections for supplying a cooling fluid to

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said condenser, a vent leading from the upper end of the compartment about said condensing surface, and cooling jacket means for said vent communicating with one of said cooling fluid connections.

2. A fractionating column head, comprising a chamber and a condenser including a generally cylindrical condensing surface projecting into said chamber at its upper end, said condensing surface being tapered at its lower end, a capillary tube having its lower end opening at said tapered lower end of said condensing surface, said capillary tube having its upper end extending outside of said condensing surface to connect to a source of lowered pressure, and means for supplying a cooling fluid to said condenser for cooling said condensing surface,

3. A fractionating column head, comprising a chamber, and a condenser including a closed condensing surface within said chamber, said condensing surface being tapered at the lower end at which end it has an opening, a capillary tube within said condenser and having its lower end communicating with and sealed to the walls defining said opening, said capillary tube having its upper end extending outside of said condenser to connect to a source of lowered pressure, and means by which a cooling medium may be brought into contact with said condensing surface for cooling the same.

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