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HIGH VACUUM FRACTIONATING STILL

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Fig. 1.

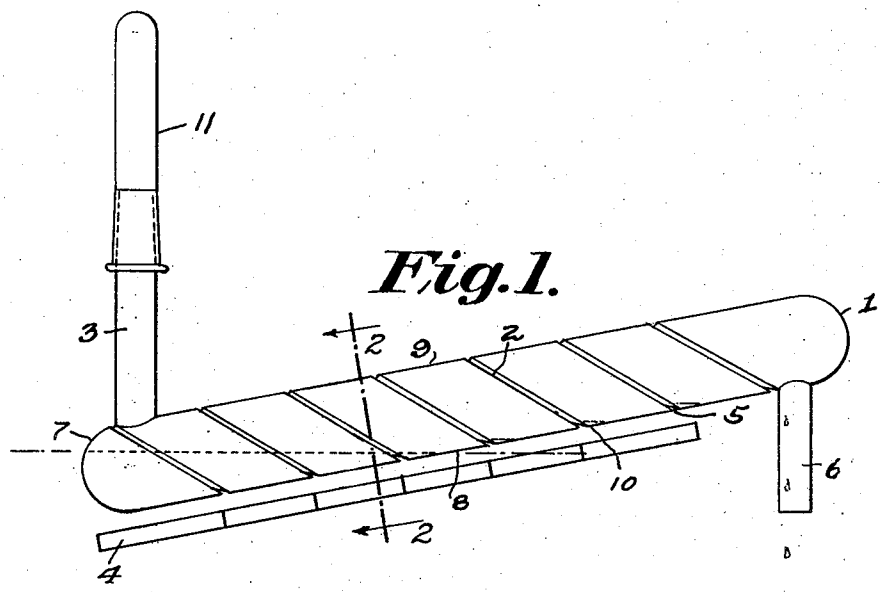
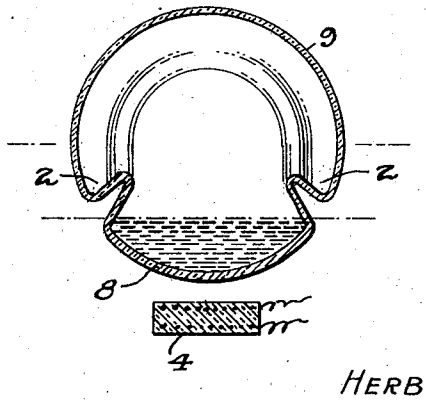


Fig. 2.



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HIGH VACUUM FRACTIONATING STILL

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4 Claims. (Cl. 202—205)

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

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The invention herein described may be manufactured and used by or for the Government of the United States for governmental purposes without the payment to us of any royalty thereon in accordance with the provisions of the act of April 30, 1928, (Ch. 460, 45 Stat. L. 467).

This invention relates to improved methods and means for distillation, and particularly to methods and means for distilling at pressures approaching an absolute vacuum.

The principal object of this invention is to provide a method and means enabling distillation in a plurality of stages within a single enclosure wherein a material being distilled from a lower film pool area condenses on a condensing surface and flows by gravity to a higher film pool area, with each successive higher pool communicating for gravity overflow to the next adjacent lower film pool area when the liquid level in the higher pool exceeds that point at which a shallow layer of a predetermined thickness exists in said higher pool providing for forward movement of condensate followed by revaporization with concurrent back flow of those portions of liquid which in each succeeding film pool area fail of revaporization.

One form of the invention is illustrated in the accompanying drawing, in which:

Fig. 1 is a vertical section or phantom view of the apparatus; and

Fig. 2 is a transverse section of the same along the axis 2—2.

In the drawing the cylinder 1 is placed at a suitable angle, such as 15° with the horizontal and is provided with a series of channels 2 of suitable shape to collect liquid condensate on the inner, upper and wall surfaces and direct its flow. These channels are disposed at such an angle that liquid flowing therein is carried forward in the cylinder despite the inclination of the cylinder in the opposite direction. The end of the cylinder 1 to which the reference numeral 1 is applied is hereinafter referred to as being forward while the portion to which reference numeral 7 is applied is to the rear. The uppermost channel empties into the duct 6 through which condensate is removed from the system. The floor of the tube is provided with a plurality of ridges 5 placed just back of the respective channels 2. The cylinder is charged through the opening 3, which is provided with a vacuum-tight closure 11.

In operation a suitable charge is placed in the cylinder and after the closure 11 is applied to the opening 3, a high vacuum is attained within

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the cylinder by means of a suitable pumping arrangement connected to the duct 6, or at any other suitable position. Heat is applied by means of electric or other heaters 4 which are arranged to be controlled in small sections.

Liquid is vaporized from the surface of the charge at any point such as 8, and rises to the roof of the cylinder 1 where condensation takes place at any point such as 9. The condensed liquid flows down the inner surface into one of the channels 2 in which it flows forward to a point 10 on the floor of the cylinder in advance of the point 8 of its origin. It is retarded in its backward flow by the ridges 5. The condensate in its forward and downward flow covers the major portion of the lower surface of the cylinder as a film which gradually flows backward, collecting in a pool restrained by the rearward ridge. Liquid is vaporized from the surface of this film pool area.

At point 10 a lesser quantity of heat is applied, causing part of the liquid to revaporize while a part of the remainder flows over the ridge and down the floor of the cylinder to the next ridge and on to the main charge.

Behind each of the ridges 5 there is formed a film pool area from which liquid constantly distills, and after condensing, moves forward. The portion not vaporized increases in volume and constantly overflows toward the main charge in the rear of the still. Each pool is continually replenished by distillation forward and reflux backward and both of these are controlled by the quantity of heat applied to each of the film pool areas.

In the above description and drawing, the ridges 5 are indicated as being immediately rearward of the channels 2 that deliver liquid upstream of the respective ridges. However, it is contemplated that the ridges 5 may be placed downstream from the point at which the respective channels 2 deliver liquid to the pools formed in part by the respective ridges so that the liquid can spread itself out in a thin layer in its backward flow permitting the lighter fractions thereof to be driven off while the heavier fractions are allowed to flow backward until they engage the respective ridges, and ultimately overflow therefrom to the next adjacent downstream pool.

It is also to be understood that instead of using the generally cylindrical still shown in the drawing, that flat surfaces may be provided as condensing surfaces and evaporating surfaces so long as the material being distilled from a lower film pool area condenses on a condensing sur-

face and flows by gravity to a higher film pool area with each successively higher pool communicating for gravity overflow with the next adjacent lower film pool area.

In fabricating a still of the type shown in the drawing, the still cylinder may be die cast, or blown of glass in a two or three part mold similar to a bottle mold, or fabricated in any other appropriate manner or from any other appropriate material.

A practical embodiment of the instant invention employing a glass still tube having an approximate mean inner diameter of 4 centimeters and a length of approximately 25 centimeters has been successfully used to fractionate marihuana extracts. In operation, a charge of acetylated marihuana oil was placed in the still through the opening 3 and the vacuum tight closure 11 consisting of a tapered ground tube was applied and the still was evacuated through duct 6 by a mercury vapor pump backed up by a high vacuum mechanical pump. A thermometer, the bulb of which was bathed in the charge in the lowermost pool, extended into the closure tube 11. After the charge had been "de-gassed" for several hours and the pressure within the still had been reduced to a near perfect vacuum as reflected by readings taken on a McLeod gauge connected to the system, heat was applied by electric heater sections (at 4) to effect distillation and pumping was continued during distillation. The various fractions of the distillate coming from the still (down the duct 6) were caught in successive containers sealed into the system, provision being made to change the containers into which the distillate was led by positioning a magnet outside of the sealed system.

The pressure as read on a McLeod gauge connected with the still through about 30 inches of glass tubing was within the range of from 1.2×10^{-4} to 7.7×10^{-5} millimeters of mercury, and the temperature of the liquid in the lowermost pool ranged from 130° C., to nearly 200° C. The top surface of the still tube was open to the ambient atmosphere within the laboratory and was thus cooled below the temperatures of the liquid within the pools. The efficiency of fractionation of the new still was interpreted on the basis of the refractive indices and of the optical rotations of the fractions obtained therefrom as compared to the indices of fractions obtained from a molecular pot still. The physiological potency of the fractions obtained from the new still was compared with the physiological potency of fractions obtained from a molecular pot still.

As determined by both the refractive index comparison and the physiological potency comparison, the separation obtained by the new still appeared to be far superior to that obtained by a single stage molecular pot still.

We claim:

1. A multiple stage still comprising a generally cylindrical inclined tube having a plurality of walls extending from the bottom of said tube generally perpendicular to its axis, said walls being of such height as to provide a plurality of adjacent shallow film pool areas for containing a distilland with each successive pool progressively higher than its predecessor, means for creating a near perfect vacuum within said tube, means for evaporating distilland from said film pool areas, condensing surfaces overlying said film pool areas and means for directing the condensate derived from a pool to flow by gravity to the next higher film pool area.

2. A multiple stage high vacuum still comprising means forming a plurality of heated distillation pools communicating for gravity flow from pool to pool in a given direction, and means including surfaces overlying said respective pools adapted to condense and deliver by gravity condensed distillate from the respective pools to the adjacent upstream pools respectively.

3. A system in accordance with claim 2 in which the condensed distillate is delivered to the respective upstream pools at points closely adjacent to the portions thereof from which liquid flows to the next adjacent downstream pools.

4. A still comprising a plurality of stages, each including a pool-forming enclosure providing an evaporating surface, and each including a superior located area providing a condensing surface, each of the pool-forming enclosures located at different levels, the higher pool-forming enclosures communicating for gravity flow to a succeeding lower pool-forming enclosure, and means for directing condensate condensed on said superior located condenser areas to flow by gravity to a higher pool-forming enclosure than that from which said condensate was evaporated, whereby the more readily distillable fractions are carried forward to higher levels with concurrent backflow of those portions which in each succeeding pool-forming enclosure fail of revaporization.

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