Dec. 10, 1940. V. VOORHEES 2,224,621

HIGH VACUUM DISTILLATION

Filed June 20, 1938

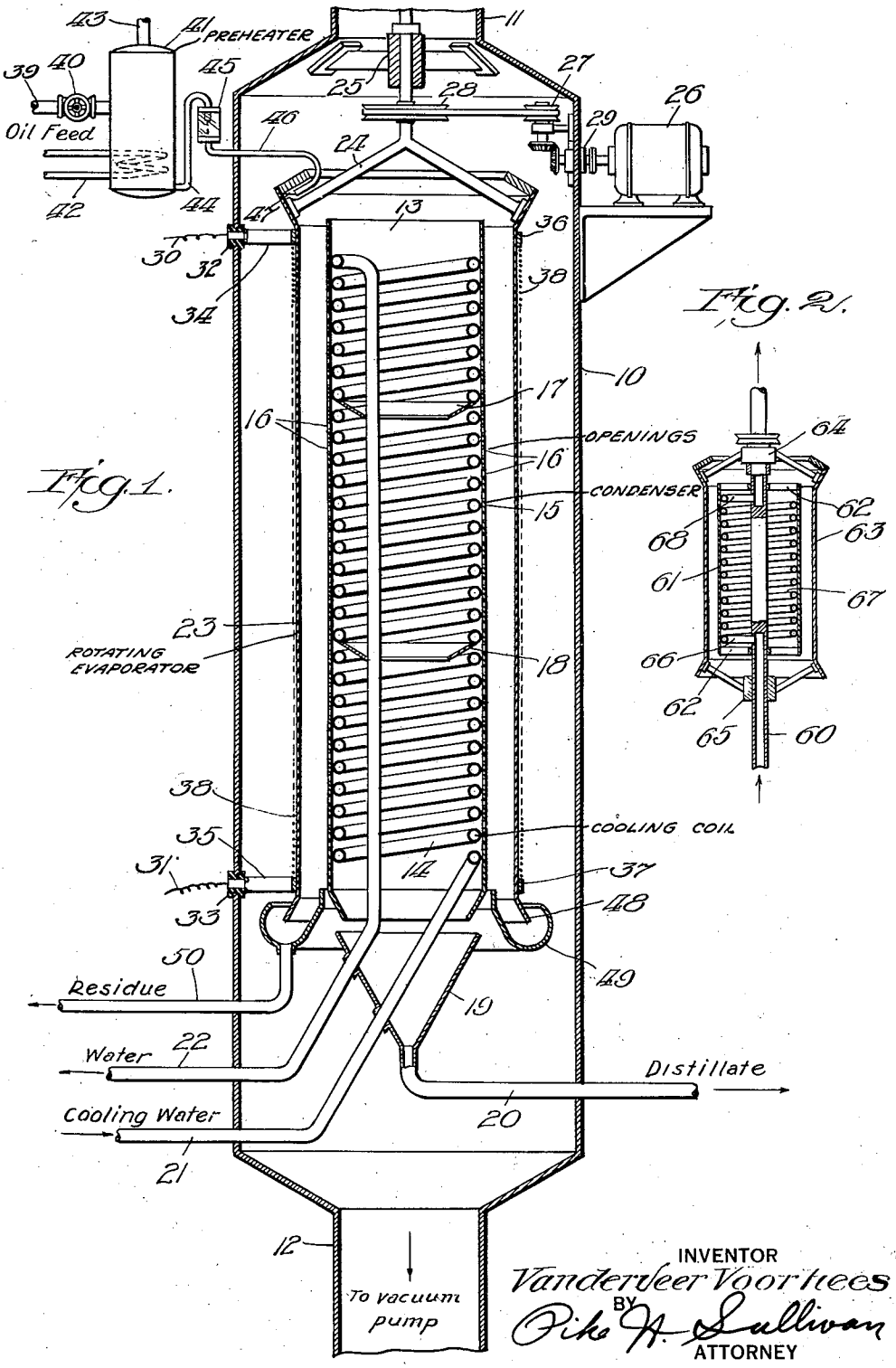


Fig. 1.

Fig. 2.

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Patented Dec. 10, 1940 2,224,621

UNITED STATES PATENT OFFICE

2,224,621

HIGH VACUUM DISTILLATION

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Application June 20, 1938, Serial No. 214,682

19 Claims. (Cl. 202—52)

This invention relates to a process and apparatus for distilling oils, and particularly for distilling heavy high boiling petroleum oils and lubricating oils. One object of the invention is to distill high boiling substances under high vacuum without decomposition. Another object is to distill high boiling substances under so-called "molecular distillation" conditions without ebullition and without entrainment and contamination of the distillate by the undistilled oil. Still another object of the invention is to provide a means for carrying out molecular distillation at lower pressures than heretofore commercially feasible, thereby permitting distilling a larger proportion of the high boiling material than previously practicable.

The invention is illustrated by a drawing in which Figure 1 shows in detail a diagrammatic form of the apparatus and Figure 2 shows an alternative structural arrangement of the evaporating and condensing surfaces in schematic form.

Referring to the drawing, Figure 1, a shell 10 is connected to an exhausting vacuum pump or pumps not shown by means of large vacuum connections 11 and 12. Alternative or supplementary connections to the exhausting means may be made on other parts of the shell, for example to the sides thereof. Within the gas tight shell 10 is located stationary condenser element 13 suitably formed of a perforated cylinder provided with a cooling coil 14 in heat conductive contact therewith. The cooling coil, for example, may be a copper tube spiral soldered to the interior surface of the condenser cylinder. The surface 15 of the condenser 13 is provided with openings 16 to permit free passage of gases and liquids from the exterior to the interior thereof. Collector baffles 17 and 18 serve to direct liquid condensate away from the interior surface of the condenser and into the receiving funnel 19 whence the liquids flow by line 20 to suitable receiver not shown. Connections 21 and 22 serve to supply cold water, oil, brine or other suitable cooling fluid to the coil 14. The direction of flow of the cooling fluid may be either to the top of the condenser or to the bottom.

Surrounding condenser 13 is evaporator 23 which is supported by spider 24 and bearing 25 providing for free rotation about its axis. Power for rotating cylinder 23 is provided by motor 26 through the geared pulley 27 and belted pulley 28. Obviously, various other means may be employed for providing rotation. In the arrangement shown a packing gland 29 prevents ingress of air through the shell 10 to the evacuated space within, although I may mount the motor inside casing 10 and thus avoid any possibility of air leakage whatever.

Heat is supplied to the surface of evaporator 23 preferably by electrical induction heater or electrical resistance winding, the latter being illustrated in the drawing. A current of electricity supplied by lead wires 30 and 31 passes through the shell 10 by insulated conductors 32 and 33. Brushes 34 and 35 bearing on suitable collector rings 36 and 37 conduct the electric current to resistance winding 38 which is suitably disposed along the outer surface of evaporating cylinder 23 to provide the heat necessary for distilling oil or other liquid on the interior surface thereof. Additional electrical connections may be provided to facilitate regulation of the heat supplied to evaporator 23. Thus separate coils may be provided at intervals along the surface of the evaporator to separately control the temperature in different sections as indicated by suitable temperature indicating means not shown. Thus the temperature may be gradually increased as one progresses from the upper end to the lower end of the evaporator 23 or, if desired, the temperature may be held substantially constant throughout.

The oil to be distilled is supplied by line 39, controlled by valve 40 leading to preheater chamber 41. The oil may be preheated in chamber 41 by heating element 42 to a suitable temperature well below its decomposition point, for example 500 to 650° F. in the case of heavy mineral oils. Any vapors evolved from the oil in the preheater 41 are exhausted by line 43 connected to a vacuum pump not shown. It is preferred to exhaust the preheater 41 to a pressure as low as that within the shell 10.

From 41 the oil flows by line 44 to sight feed 45 and thence by line 46 to the upper end of evaporator 23. The oil is suitably introduced into the evaporator by allowing it to flow into channel member 47 where it collects by virtue of the action of the centrifugal force resulting from the rotation of evaporator 23 hereinbefore mentioned. From channel 47 the oil flows in a uniformly thin film downward over the inner surface of evaporator 23, finally passing out at the bottom over lip 48. Trough 49 collects the residual oil from which it is conducted by outlet line 50.

Various other means may be employed for supporting the evaporating and condensing surfaces 23 and 14. For example, in Figure 2 the hollow stationary shaft 60 supports condensing surface 61 by spiders 62, and evaporating surface 63 is rotated thereabout on bearings 64 and 65. Cooling water for the condenser is supplied through the hollow shaft 60 entering at one end and passing by connection 66 to coil 67 and thence by connection 68 to the supporting shaft 69 and out at the other end.

Referring again to Figure 1 the inner surface of the revolving evaporator 23 is preferably pol-

ished to reduce the amount of heat lost by radiation to the condenser 13. The polished surface may be obtained by plating with a corrosion resistant metal, for example, chromium, platinum, gold, aluminum, silver, copper, etc., or the cylinder 23 may be constructed of a suitable corrosion resistant alloy, such as stainless steel. The exterior surface 15 of the condenser 13 is likewise preferably polished to prevent absorption of radiant heat. The surface of the evaporator 23 may be truly cylindrical throughout its length or, if desired, it may be slightly conical with the apex in either direction, but preferably toward the top, thus providing more rapid flow of liquid downward along the surface. In that case, the condenser 13 should likewise be conical to correspond therewith.

The distance separating the condensing surface 15 from the evaporating surface 23 is preferably very short, in order to provide a minimum distance for evaporated oil molecules to travel from the heated surface to the cold condensing surface. It is preferred to adjust the surfaces so that the distances between the evaporator and condenser approximate the mean free path of the oil molecules under the conditions of temperature and pressure prevailing. However, much larger distances may be employed, for example, up to fifty or even one hundred times the mean free path of the molecules. Thus, distances of 0.05 to 0.4 inch are contemplated.

It is not necessary to rotate the evaporating element 23 of my vacuum distillation apparatus at very high speeds in order to maintain a uniform film of oil on the interior surface thereof. For example, I may operate with speeds of only 200 to 600 revolutions per minute. At such speeds of rotation the oil is distributed in a uniform film over the inner surface making it possible to adjust the space between the evaporator and the condenser very closely and accurately without contamination of the distillate on the condenser occurring as a result of physical contact between irregularities on the distilling surface. The rotating motion also serves to provide agitation of the oil, thus exposing continually new film surface to evaporation.

Gases dissolved in the oil and any vaporous decomposition product thereof which may be liberated at the distilling surface find a ready egress through the interstices of the condensing surface 15 hereinbefore mentioned and therefore no possibility exists for substantial pressures to build up along the evaporating surface and impede the distillation. Gases and vapors entering the interior of the condenser 13 rapidly find their way to the ample outlets 11 and 12 connecting to the vacuum pump. The vacuum is preferably supplied by vapor pumps supported by high pressure steam ejectors or mechanical pumps. Pressures of the order of 0.1 to .00001 mm. mercury may be employed, a suitable pressure for most purposes being 0.01 mm. of mercury. One of the important advantages of my vacuum distillation apparatus lies in the equalization of pressure therein by use of unobstructed gas passages from evaporating surface to vacuum pump, thus providing substantially the same pressure at the evaporating surface as at the vacuum pump.

If desired, I may rotate the evaporating element of my apparatus at sufficient speed to build up by centrifugal action, increased pressure within the oil film adjacent the surface of the evaporator 23 thus retarding ebullition of the oil and permitting the use of somewhat higher temperatures without entrainment difficulty. By this means the development of a vapor phase within the oil film in contact with the metallic heating surface is avoided and superheating of the body of oil within the oil film, above its boiling point at the low pressure prevailing, is made possible.

Although I have described my invention with respect to certain embodiments thereof it should be understood that various modifications may be employed without departing from the spirit thereof. Thus I may employ in a series two or more distillation units, as described, conducting the oil from one unit to the next to produce a series of fractions of increasing molecular weight. I may also segregate the distillate from succeeding sections of the condenser in a single unit for the same purpose. I may charge the apparatus with crude oil residue, for example, a 40% residue from Pennsylvania or Mid-Continent crude oil. I may also apply my distillation process to distillation of other materials, such as coal tars, vegetable and animal oils, fats and waxes, for example, degras, olive oil, chaulmoogra oil, tung oil, etc. I may also apply my distillation method to the recovery of vitamines from various products, such as fish liver oils, e. g., menhaden, cod, halibut liver oils, irradiated fats, etc. In general, the method may be applied to the distillation of any liquefiable substances whose separation is desired without decomposition. The method is particularly valuable for the distillation of lubricating oils where loss of viscosity from decomposition is a serious objection to the use of ordinary distillation methds.

I claim:

1. The process of distilling liquids under high vacuum without ebullition which comprises maintaining a cooled, substantially cylindrical, condensing surface positioned to permit withdrawal of condensate and surrounding said condensing surface in close proximity thereto and concentric therewith, a heated, rotatable, substantially cylindrical, evaporating surface, introducing a stream of said liquid at a point on the interior of said evaporating surface, rotating said evaporating surface at a speed adapted to subject the liquid to a sufficient centrifugal force to uniformly distribute it over the surface thereof, collecting and withdrawing distillate from said condensing surface and discharging unevaporated liquid from said evaporating surface at a point remote from said point of introduction.

2. The process of claim 1 wherein said distillation is conducted under a pressure of 0.1 to .00001 mm. mercury pressure.

3. The process of claim 1 wherein the distillation is conducted under a low pressure of the order of .01 mm. mercury, gases evolved from said liquid undergoing distillation are permitted to pass through openings in said cylindrical condensing surface leading to the interior thereof and thence are discharged from the end thereof.

4. The process of claim 1 wherein said liquid is a petroleum lubricating oil.

5. The process of claim 1 wherein said liquid is an animal fat.

6. In a molecular distillation process wherein a liquid is distilled without boiling by maintaining a heated surface thereof in close proximity to a cooled, condensing surface at a low pressure of the order of 0.01 mm. mercury, the improvement comprising distributing said liquid on the interior surface of a heated revolving cylinder, condensing the vapors on a cooled, condensing

surface positioned to permit withdrawal of condensate and located within said revolving cylinder and in close proximity to the surface thereof and causing said cylinder to revolve at a rate of speed sufficient to subject the said liquid retained therein to centrifugal force sufficient to retard ebullition of said liquid.

7. In a molecular still wherein a crude, liquid material is distributed over a heated, evaporating surface in close proximity to a cooled, condensing surface, the improvement comprising a gas-tight shell, a connection leading from said shell to a gas exhausting means, a rotatable, heated, cylindrical, evaporating surface within said shell, a cooled, cylindrical, condensing surface positioned to permit withdrawal of condensate and located within said evaporating surface and in close proximity thereto, means for rotating said evaporating surface, means for introducing a stream of liquid onto said evaporating surface within the space between it and the said condensing surface, means for withdrawing unevaporated residue from said evaporating surface and means for withdrawing distillate from said condensing surface.

8. A molecular still as described in claim 7 further characterized in that the evaporating and condensing surfaces are vertical.

9. In a molecular still wherein a crude, liquid material is distributed over a heated, evaporating surface in close proximity to a cooled, condensing surface, the improvement comprising a gas-tight shell, a connection leading from said shell to a gas exhausting means, a rotatable, heated, evaporating surface within said shell, a cooled, condensing surface positioned to permit withdrawal of condensate and located in close proximity to said evaporating surface and nearer to the axis of rotation of the evaporating surface than is the evaporating surface itself, means for rotating said evaporating surface, means for introducing a liquid onto said evaporating surface within the space between it and said condensing surface, means for discharging unevaporated residue from said evaporating surface and means for collecting and withdrawing distillate from said condensing surface.

10. The apparatus of claim 9 wherein means are provided for heating said liquid at a low pressure on the order of .01 mm. mercury before introducing it onto said evaporating surface and means for maintaining a pressure within said shell of the order of .01 mm.

11. The apparatus of claim 9 wherein said evaporating surface is heated by electrical means.

12. In a molecular still wherein a crude liquid material is distributed over a heated, evaporating surface in close proximity to a cooled, condensing surface, the improvement comprising a cylindrical, gas-tight shell, a substantially cylindrical, rotatable, heated, evaporating surface within said shell, a substantially cylindrical condenser providing a cooled, condensing surface positioned to permit withdrawal of condensate and located within said evaporating surface, means for introducing a liquid onto said evaporating surface between it and said condensing surface while said evaporating surface is in rotation, means for withdrawing unevaporated liquid from said evaporating surface, means for withdrawing distillate from said condensing surface, means for exhausting gases from said shell, and means for providing a substantially unobstructed path for gases from said evaporating surface through said condensing surface thence through the end of said cylindrical condenser and through said gas-tight shell to said gas exhausting means.

13. In a molecular distillation process wherein a liquid is distilled from an evaporating surface to a condensing surface without ebullition, the improvement which comprises maintaining the evaporating liquid under a centrifugal pressure in a direction away from the condensing surface.

14. The process of claim 13 wherein said distillation is conducted under a pressure of 0.1 to .00001 mm. mercury pressure.

15. The process of claim 13 wherein said liquid is a petroleum lubricating oil.

16. The process of distilling liquids under high vacuum without ebullition which comprises introducing a stream of the liquid upon a rotatable, heated, evaporating surface which is in close proximity to a cooled, condensing surface positioned to permit withdrawal of condensate and located so that a centrifugal force produced by rotation of the evaporating surface operates in a direction away from said condensing surface, maintaining the space between the said surfaces under a high vacuum, rotating said evaporating surface at a speed adapted to subject the liquid to a sufficient, centrifugal force to uniformly distribute said liquid over the surface thereof, and collecting and withdrawing distillate from said condensing surface.

17. In a molecular still wherein a crude, liquid material is distributed over a heated, evaporating surface in close proximity to a cooled, condensing surface, the improvement comprising a gas-tight shell, a connection leading from said shell to a gas exhausting means, a rotatable, heated, cylindrical, evaporating surface within said shell, a cooled, cylindrical, condensing surface positioned to permit withdrawal of condensate and located within said evaporating surface and in close proximity thereto, means for rotating said evaporating surface, means for introducing a stream of liquid onto said evaporating surface within the space between it and the said condensing surface, and means for withdrawing distillate from said condensing surface.

18. In a molecular still wherein a crude liquid material is distributed over a heated, evaporating surface in close proximity to a cooled, condensing surface, the improvement comprising a gas-tight shell, a connection leading from said shell to a gas exhausting means, a rotatable, heated, evaporating surface within said shell, a cooled condensing surface positioned to permit withdrawal of distillate and located in close proximity to said evaporating surface and nearer to the axis of rotation of the evaporating surface than is the evaporating surface itself, means for rotating said evaporating surface, means for introducing a liquid onto said evaporating surface within the space between it and said condensing surface, and means for collecting and withdrawing distillate from said condensing surface.

19. The apparatus of claim 18 wherein means are provided for heating said liquid at a low pressure on the order of .01 mm. mercury before introducing it onto said evaporating surface and wherein means are also provided for maintaining a pressure within said shell of the order of .01 mm. mercury.

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