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VACUUM APPARATUS

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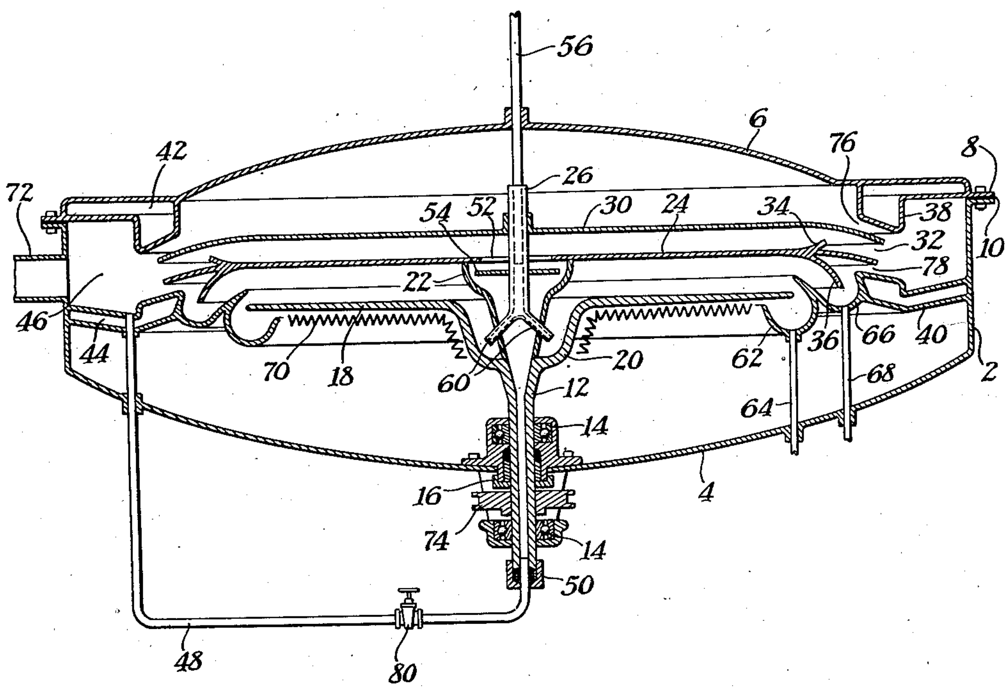

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VACUUM APPARATUS

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This invention relates to improved high vacuum stills, and particularly self-pumping high vacuum stills. The invention also pertains to an improved high vacuum pump.

Although heat losses have heretofore been a real problem in connection with high vacuum, and particularly high vacuum unobstructed path stills, the problem has not heretofore been solved. It is, of course, known to regenerate the heat of condensation in ordinary stills but such methods have not been applicable to high vacuum stills. Also, it has heretofore been proposed to utilize the vapors of a molecular still to effect a pumping action. However, in this prior art suggestion the heat losses were the same as in ordinary high vacuum or molecular stills. It was the motion of distilling molecules rather than the heat contained therein which was slightly used to effect a pumping action. Also it has been realized that great heat loss takes place by radiation from the hot vaporizing surface to the cold condensing surface of a high vacuum, unobstructed path still.

This invention has for its object to provide improved vacuum distillation apparatus whereby the above difficulties can be avoided. Another object is to provide high vacuum unobstructed path distillation apparatus in which the heat losses are greatly reduced. Another object is to provide high vacuum distillation apparatus wherein the hot vaporizing surface and the cool condensing surface are located opposite to each other but wherein heat losses common to such conditions or constructions are substantially avoided. A still further object is to provide an improved self-pumping high vacuum still wherein the heat of condensation is utilized to effect evacuation of the still. Another object is to provide improved vacuum pumps. Other objects will appear hereinafter.

These and other objects are accomplished in accordance with my invention which includes high vacuum distillation apparatus wherein the heat of radiation from the vaporizing plate and/or the heat of condensation is utilized to vaporize the working fluid in the evacuating ejector or condensation pump. The invention also includes improved pumps wherein the pump liquid is very effectively vaporized by passage over a heated surface in a thin film by centrifugal force.

In the following examples and description I have given several of the preferred embodiments of my invention but it is to be understood that these are set forth for purposes of illustration and not in limitation thereof.

In the accompanying drawing is shown a vertical section of a preferred form of my invention, comprising a self pumping high vacuum unobstructed path still. Referring to the drawing, numeral 2 designates a gas-tight casing provided with an integral bell shaped base 4 and a removable bell shaped top 6. A flange 8 is provided with intermediate gasket 10 to enable gas tight assembly of 6 with the still casing 2. Numeral 12 designates a shaft rigidly housed in bearings 14 and packed gland 16. Numeral 18 designates a circular plate having a depressed cup-shaped portion 20 at the center which is integral with hollow shaft 12. Numeral 22 designates a cup-shaped extension of shaft 12 which is integral with circular plate 24 and supports and maintains plate 24 rigidly in the position illustrated. Numeral 26 designates a hollow shaft the axis of which is in line with the axis of hollow shaft 12 and which is integral with the cup-shaped portion 22 of shaft 12 at breach 60.

Numeral 30 designates a circular plate substantially parallel with plate 24 and in spaced relation thereto. The peripheries of plates 24 and 30 are curved downwardly and upper plate 30 has a somewhat greater curvature so that the two plates cooperate at their peripheries to form an annular nozzle 32. Plate 24 is provided with circular projections 34 and 36 as illustrated. Bell shaped top 6 is provided at its periphery with an integral circular projection 38, and casing 2 is provided with a similar annular projection 40. Projections 38 and 40 are so formed that they will in combination with nozzle 32 form an ejector or condensation pump nozzle. Cooling fluid is circulated through the hollow portions 42 and 44 of projections 38 and 40 respectively.

Numeral 46 designates an annular chamber communicating with the exhaust from nozzle 32. Numeral 48 designates a conduit connected to the lower portion of 46 and leading to the hollow shaft 12 to which it is connected by a packed gland 50. Numeral 52 designates a hole in the center of plate 24 and numeral 54 designates a circular baffle integral with shaft 26. Numeral 56 designates a stationary shaft protruding into shaft 26. Numeral 60 designates a double or breach opening at the base of shaft 26. Numeral 62 designates an annular gutter into which circular plate 18 protrudes. Numeral 64 designates a withdrawal conduit connected to gutter 62. Numeral 66 designates a similar annular gutter into which protrude extension 36 of plate 24. Numeral 68 designates a conduit connected to gutter 66. Numeral 70 designates an electrical heating unit for vaporizing plate 18. Numeral

72 designates a conduit connected to a fore or backing pump (not shown).

In operating the apparatus illustrated a partial vacuum is produced in the system by a pump (not shown) connected to conduit 72. Heating element 70 is put into operation in order to heat circular vaporizing plate 18 to distillation temperature. A suitable vaporizable pump fluid is introduced into annual chamber 46 and liquid to be distilled is introduced into the depressed cup 20 through stationary conduit 56, revolving conduit 26 and breach 60. Shaft 12 is then rotated by means of force applied to pulley 74. As a result, vaporizing plate 18 and plates 24 and 30 rotate as one integral unit. Liquid to be distilled flows downward through conduit 26 and breach 60 into cup 20 and is then caused to flow by centrifugal force over the top of vaporizing surface 18 in the form of thin film. During the passage thereover distillation takes place. Undistilled residue is thrown into annular gutter 62 and is withdrawn from the still through conduit 64. Vapors generated during distillation pass directly to condensing surface 24 where they are condensed and thrown by centrifugal force to projection 36 and then into gutter 66 from which the condensate is withdrawn through conduit 68.

During the foregoing operations the pump fluid in 46 flows by gravity through conduit 48 upwardly through the center of shaft 12 into bell 22. It is then lifted by centrifugal force past the baffle 54 and through the circular opening 52. It then flows as a thin film over the top surface of plate 24, or in other words over that side of plate 24 away from plate 18. During passage it is heated primarily by radiation from plate 24 and also by the vapors condensing on the opposite side of plate 24. This heat is taken up by the pump fluid which is vaporized and the vapors thus generated flow by a combination of centrifugal force and their own kinetic energy through annular nozzle 32, as a jet or stream of high velocity. Gases present in the still pass into this jet stream through openings 76 and 78 and become entrained in the vapor jet and are thus forced into chamber 46. The vapors from the jet are condensed by contact with the cool walls of chamber 46 which are cooled by cooling fluid circulated through 42 and 44. Permanent or uncondensed gases are withdrawn through conduit 72 by the backing pump (not shown). In practice the still will become a little hotter than normal during the starting period and the extra radiant heat will start the pump in action, distillation will then commence and the still will cool down to its steady running temperature.

Any pumping fluid which is not vaporized on the top of plate 24 is thrown by projection 34 against the bottom of plate 30 so that the stream issuing from nozzle 32 is a relatively homogenous vapor.

The combination illustrated provides an annular or circular jet nozzle with two annular intakes thereto. The intakes are of high admittance and accordingly a condensation pump of high capacity is located in the still just where it is needed, namely, as near the place from which the gas is to be removed as is possible.

The rate of flow of the pump fluid to the plate 24 can be controlled by a valve such as shown at 80. It will be realized that the rate of introduction of pump fluid onto plate 24 will be adjusted to suit the temperature of the vaporizing surface and the rate of distillation thereon. If the rate of distillation and the temperature are low, the pump fluid should be introduced at a correspondingly low rate since if it is introduced in too great an amount, it will effectively cool condensing surface 24 but it will not be effectively vaporized by the heat of radiation and/or condensation.

The pump fluid utilized should have a boiling point which is somewhat lower under the conditions prevailing than the boiling point of the distillate to be produced in the still at the time the pump fluid is to be used. The procedure is of greatest value for the distillation of substances at high temperatures such as above 200° C. A satisfactory pump fluid for such conditions would be dipropylphthalate. The pump fluid should be one which enables a satisfactory low condenser temperature to be used but which will be substantially vaporized at said condenser temperature. However, it is not necessary to utilize a condenser which is at room temperature. With many substances it is satisfactory and indeed preferred to utilize a condenser temperature of 100° or perhaps even 150° C. or higher. If the use of high condenser temperatures is a satisfactory method of operation in connection with the substance to be distilled, a higher boiling point pump fluid can be used, and hence greater pumping efficiency will result. The following are examples of pump fluids and condenser temperatures which may be used.

Condenser temperature near—
100°, diethyl phthalate
120°, dipropyl phthalate
130°, dibutyl phthalate
150°, diamyl phthalate
180°–200°, dioctyl phthalate or dioctyl sebacate

In place of these pump fluids the methyl and ethyl esters of the fatty acids can be used. Free fatty acids such as oleic stearic and natural mixtures of free fatty acids can be used.

Many modifications can be made in the above described apparatus without departing from the spirit or scope of my invention. For instance, the condenser plates 24 and 30 need not be fastened to the same shaft as vaporizing plate 18. They can be rotated by a separate shaft. Also rotation of either or both of plates 24 and 30 is unnecessary although advantageous. If desired they can be stationary but shaped so that they have a slight slope, in which case the pump fluid would pass over the surface by gravity.

Although the invention is of particular value in connection with centrifugal stills because they have a high rate of distillation and accordingly a high heat of condensation, the invention can be utilized with advantage in connection with ordinary gravity flow stills. The pump fluid passing through conduit 48 can be caused to flow by means of a pump instead of by gravity, if desired.

The invention has the particular advantage that the extravagant expenditure of heat associated with high vacuum unobstructed path, high vacuum short path, or molecular distillation need no longer be wasted A further and important advantage is that the condensing surface is maintained at a definite temperature throughout the distillation; i. e., the temperature at which the pump fluid vaporizes. This is important in connection with the distillation of substances containing solid matter such as sterols since they are thus prevented from crystallizing and depositing on the vaporizing surface. Also, an important advantage is that the pumping is produced right in the still and right at the place where it is needed, namely immediately at or near the vaporizing zone. The still and pumping construction is also simplified so that a complete still can be directly fastened to the primary vacuum lines which are able to provide a cheap and efficient form of vacuum.

The rate of rotation is not of critical importance and in general will be between 200 and 30,000 R. P. M. per minute. Rates of 500 to 4,000 R. P. M. are usually convenient. For further details regarding the construction and operation of rotating stills reference is made to my U. S. Patents 2,210,927 and 2,210,928.

What I claim is:

1. Vacuum distillation apparatus comprising in combination a vaporizing surface, a condensing surface, means for introducing distilland onto the vaporizing surface, means for removing undistilled residue from the vaporizing surface, means for removing distillate from the condensing surface, a jet pump means for bringing the working fluid of the evacuating jet pump the intake side of which communicates with the still and the space between the vaporizing and condensing surfaces into thermal contact with the distilling vapors, whereby during operation of the still the heat from the vapors passes to the working fluid causing the working fluid to be converted into vapors which pass through the jet pump and thus entrain and remove gases from the still.

2 Vacuum distillation apparatus comprising in combination a vaporizing surface, a condensing surface separated from the vaporizing surface by substantially unobstructed space, means for introducing distilland onto the vaporizing surface, means for removing undistilled residue from the vaporizing surface, means for removing distillate from the condensing surface, an evacuating jet pump, means for exposing the working fluid of the evacuating jet the intake side of which communicates with the still and the space between the vaporizing and condensing surfaces, to the radiation of the vaporizing surface and the heat content of the distilling vapors whereby during operation of the still the heat from the vapors and heat of radiation pass to the working fluid causing the working fluid to be converted into vapors which pass through the jet pump and thus entrain and remove gases from the still.

3. Vacuum distillation apparatus comprising in combination a vaporizing surface, a condensing surface located opposite to the vaporizing surface and separated therefrom by substantially unobstructed space, means for introducing distilland onto the vaporizing surface, means for removing undistilled residue from the vaporizing surface, means for removing distillate from condensing surface an evacuating jet pump the intake side of which communicates with the system and the space between the vaporizing and condensing surfaces, the throat of said jet pump communicating with the side of the condensing surface away from the vaporizing surface and, means for introducing evacuating or pump working fluid onto that side of the condensing surface which is farthest from the vaporizing surface, whereby during operation of the still the heat of radiation from the vaporizing surface and the heat from the vapors condensing on one side of the condensing surface pass to the working fluid on the other side of the condensing surface causing the working fluid to be converted into vapors which pass through the jet pump thus causing evacuation of the still.

4. Vacuum distillation apparatus comprising in combination a vaporizing surface, a condensing surface, means for introducing distilland onto the vaporizing surface, means for removing undistilled residue from the vaporizing surface; means for removing distillate from the condensing surface an evacuating jet pump means for bringing together the heat radiated from the vaporizing surface and the working fluid of the evacuating jet pump the intake side of which communicates with a system to be evacuated, whereby during operation of the still the heat radiated from the vaporizing surface causes the working fluid to be converted into vapors which pass through the jet pump and thus entrain and remove gases from the system.

5. High vacuum unobstructed path distillation apparatus comprising in combination within a closed system, a rotatable vaporizing surface, a condensing surface separated from the vaporizing surface by substantially unobstructed space, means for introducing distilland onto the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, means for removing distillate from the condensing surface, an evacuating jet pump means for introducing a condensation pump working fluid onto that side of the condensing surface away from the vaporizing surface means for conveying condensation pump working fluid vapors from that side of the condensing surface away from the vaporizing surface to the throat of the jet pump and passages for gases in the system to flow to the intake side of the jet pump whereby during operation of the still distilland is caused to flow in a thin film by centrifugal force over the rotating vaporizing surface, distilling vapors are condensed and simultaneously give up their heat to the pump fluid on the opposite side of the condensing surface which is also heated by radiation from the vaporizing surface and thus the pump fluid is vaporized and passes through the jet pump and entrains gases within the system.

6. High vacuum unobstructed path distillation apparatus comprising in combination within a closed system, a rotatable vaporizing surface, a rotatable condensing surface separated from the vaporizing surface by substantially unobstructed space, means for introducing distilland onto the approximate center of the vaporizing surface, means for removing undistilled residue from the vaporizing surface, means for removing distillate from the condensing surface, means for introducing a condensation pump working fluid onto the approximate center of that side of the condensing surface away from the vaporizing surface an evacuating jet pump, means for conveying condensation pump working fluid vapors from that side of the condensing surface away from the vaporizing surface to the throat of the jet pump and passages for the gases in the system to flow to the intake side of the jet pump whereby during operation of the still, distilling vapors are condensed on the condensing surface and simultaneously give up their heat to the pump fluid on the opposite side of the condensing surface which is also heated by radiation from the vaporizing surface and thus the pump fluid is vaporized and passes through the jet pump causing evacuation of the still.

7 A condensation or ejector pump adapted to evacuate a closed receptacle and comprising in

combination a rotatable vapor-producing means which is rotatable about an axis so that pump fluid applied near to the axis of rotation is caused to flow in a thin film by centrifugal force radially with respect to the axis, means for heating the vapor-producing means, means for introducing pump fluid onto the vapor-producing means at a point near to the axis of rotation, means for collecting unvaporized pump fluid at the periphery of the rotatable vapor-producing means, a nozzle adapted during operation to form a jet of vapors which nozzle is so connected to to the vapor-producing means that the vapors generated by the vapor-producing means pass through the nozzle and are formed into a jet of vapors, an entraining chamber communicating with the receptacle to be evacuated and with the said nozzle which entraining chamber is adapted to convey gases from the receptacle to be evacuated into the jet of vapors formed by the said nozzle, means for condensing vapors after they have passed through the nozzle and means for returning at least part of the condensed vapors to the rotatable vapor-producing means.

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