March 14, 1939. K. C. D. HICKMAN 2,150,685

PROCESS AND MEANS FOR THE PRODUCTION OF VACUA

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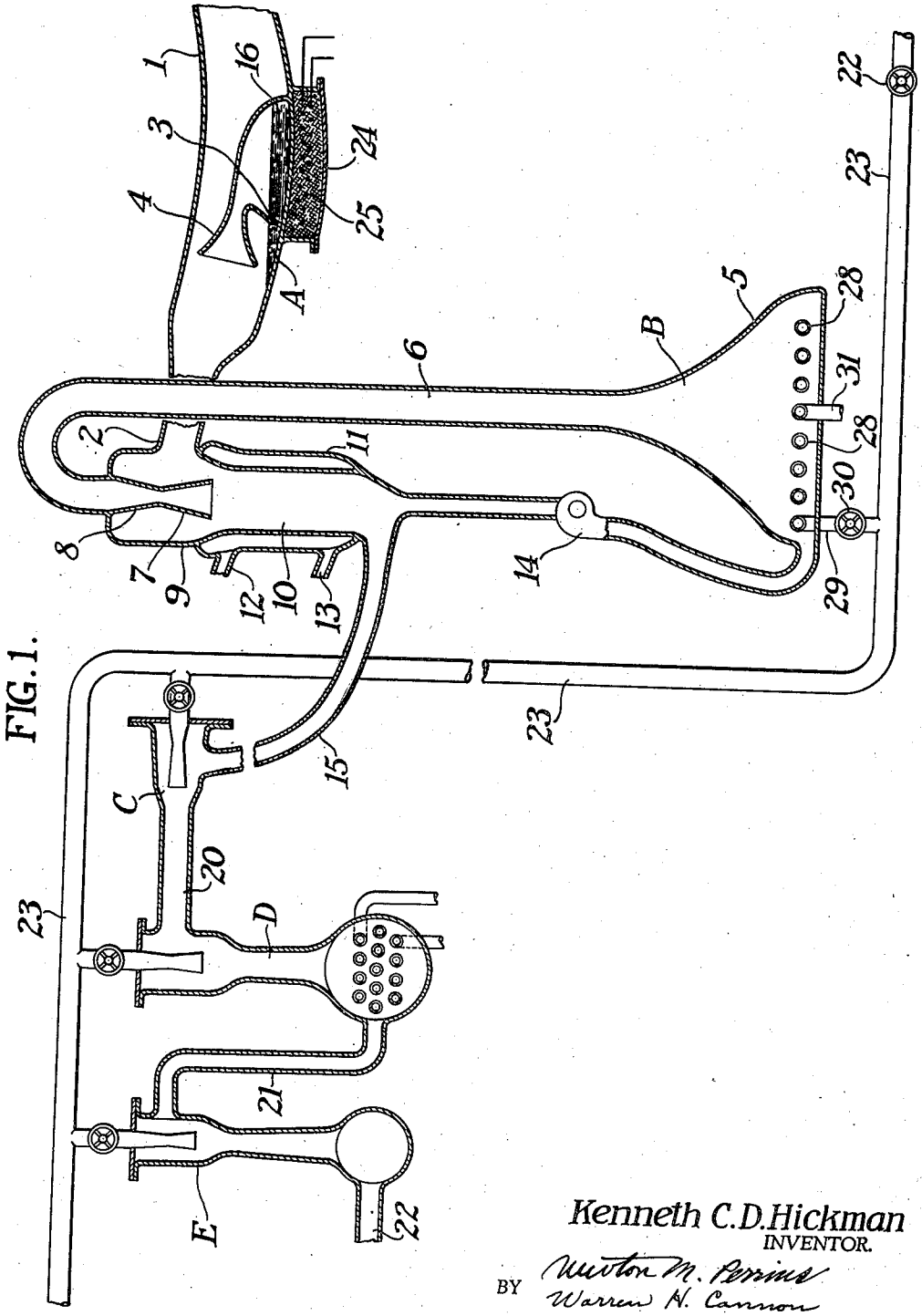


FIG. 1.

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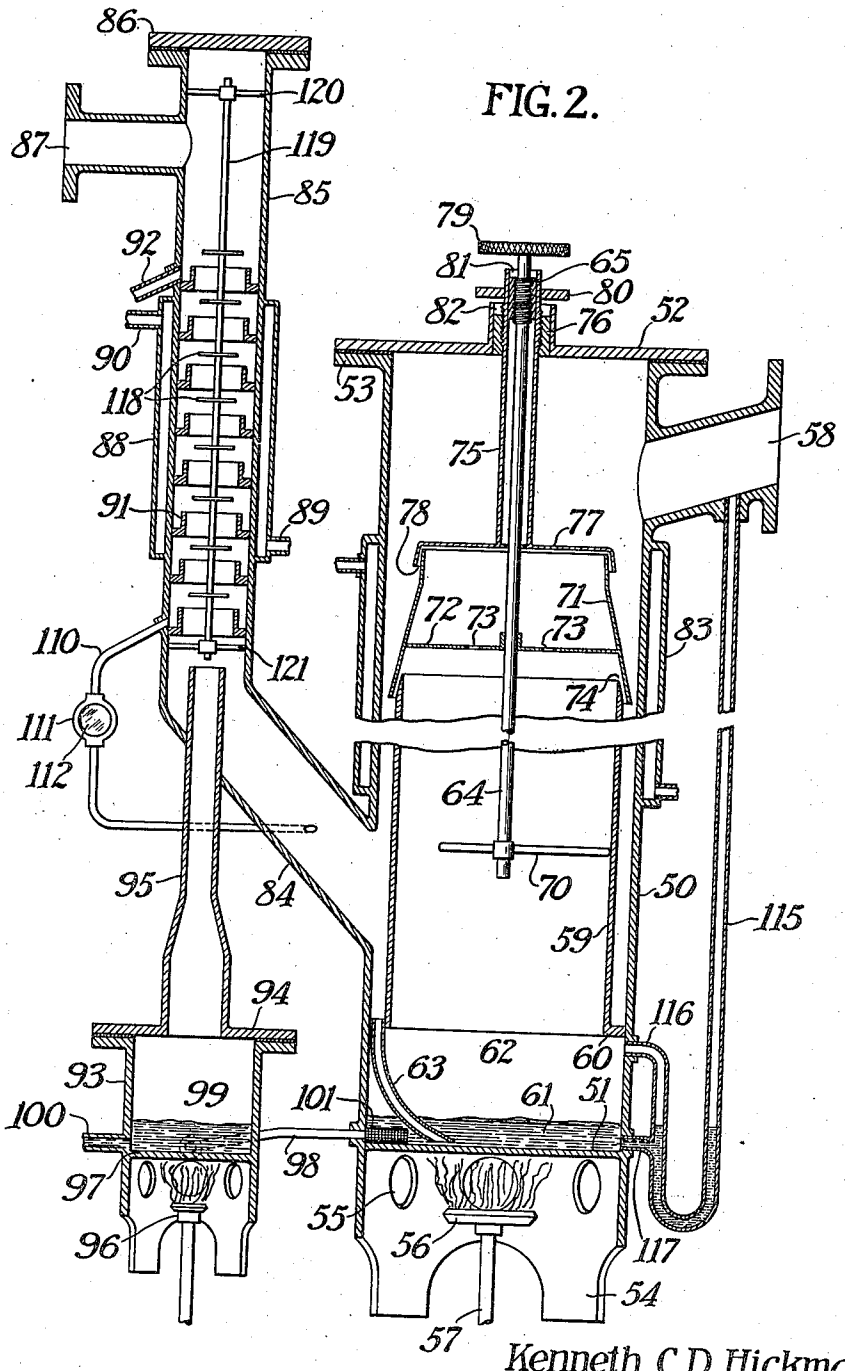
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Filed March 11, 1937 2 Sheets-Sheet 2

FIG. 2.



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Patented Mar. 14, 1939 2,150,685

UNITED STATES PATENT OFFICE

2,150,685

PROCESS AND MEANS FOR THE PRODUCTION OF VACUA

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Application March 11, 1937, Serial No. 130,359

3 Claims. (Cl. 230—101)

This invention relates to improved methods of high vacuum production and improvements in condensation or ejector pumps.

Mercury condensation pumps have been in use for a considerable number of years. Recently organic pumping fluids have been developed which have practically replaced mercury due to their higher pumping speed and ability to give a higher degree of vacuum. Mercury had the distinct advantage that it would operate against a much higher backing pressure, and in many multi-stage mercury vapor pumps efficient pumping was obtained with high backing pressures of the order of 20 mm. Ordinary mechanical vacuum pumps were entirely suitable as backing pumps for this reason. Organic pumping fluids require a very low backing pressure usually less than .1 mm. and preferably as low as about .02 mm. and below. While the condensation pumps using organic fluids will work against a slightly higher backing pressure, their pumping action becomes exceedingly erratic with backing pressures of .1–.2 mm. and at slightly lower pressures the pumping action is very slow. At backing pressures of .3 mm. the pumping action ceases entirely. This characteristic necessitates the use of specially constructed mechanical pumps giving very low backing pressures with a great loss in efficiency. These pumps do not have sufficiently high capacity for commercial high vacuum distillations and a plurality must be used further increasing the complexity and expense of operation. Another objectionable feature of organic pump fluids is their tendency to decompose with overheating caused by breaking the vacuum or stopping of the pumping operation.

A further disadvantage of such pumps, especially those of large size, is a tendency to boil dry, necessitating constant or periodic addition of fresh pumping fluid. This has been due in most cases to incomplete condensation or improper selection of pumping and condensing conditions. Another cause is that in heavy duty pumping so much gas passes through the pump that the operating vapor is carried away in the stream. When high boiler pressures are employed these effects are especially noticeable since the pump liquid is rapidly converted into more volatile materials which are withdrawn without condensation.

This invention has for its object to overcome or minimize the above deficiencies of pumps filled with organic pumping fluids. Another object is to provide an improved method which will enable organic pump fluids to be used with backing pressures obtainable with ordinary mechanical pumps or high velocity steam ejectors. Another object is to provide a pump using organic liquids which will efficiently operate against a backing pressure considerably higher than that which has heretofore been necessary with such pumps. A further object is to provide a multi-stage pump employing organic liquids which will give a vacuum as high as that heretofore obtainable with organic fluids and which will efficiently operate against backing pressures substantially in excess of .2 mm. without substantial decomposition. A further object is to provide a method of heating the pump fluids which will avoid substantial decomposition. Another object is to provide a pump in which loss of pumping fluid resulting in the pump boiler becoming dry, is avoided. Other objects will appear hereinafter.

These objects are accomplished by the herein described invention which in its preferred embodiment comprises employing considerably higher boiler pressures in an organic condensation pump than has heretofore been used in pumps of this type. Sufficient kinetic energy is thus imparted to the vapors of the organic pumping fluid that a higher backing pressure can be overcome. Substantial decomposition at the higher temperatures necessary to produce the higher pressure is avoided by employing an organic pump fluid which has a higher vapor pressure than true low vapor pressure condensation pump fluids heretofore used. Where sufficiently high vacua is not produced by such a pump it may be used in series with a condensation pump employing a true low vapor pressure organic pump fluid. By operating in this manner, a steam ejector can be used for backing purposes resulting in greatly increased efficiency of the pumping unit. Loss of pumping fluid resulting in the pump boiling dry is prevented by fractionating the pump vapors allowing only the light ends to be removed by the backing pump.

Condensation pumps filled with organic pump fluids have been unsatisfactory against high backing pressures due to the ease with which such organic compounds are decomposed. Organic condensation pump fluids have a very low vapor pressure and this fact accounts for the high vacuum which they give as compared with mercury when cooled traps are not used. This same property is also responsible for the ease of decomposition and inability to produce a high vapor pressure of pump fluid, especially at pressures easily obtained with ordinary mechanical pumps or steam ejectors. While a mercury condensation pump will oper-

ate against backing pressures of 15–20 mm. this is due to the fact that a high boiler vapor pressure of mercury such as 250 mm. can be used without fear of decomposition. Any attempt to produce a boiler pressure approaching this value with ordinary organic condensation pumps would result in complete decomposition of the pumping fluid. As a result organic fluid pumps have been operated under very low vapor or boiler pressures and published descriptions of their use are replete with warnings to avoid vapor or boiler pressures above the extremely low value of 1.5 cm. of the pumping fluid (about 1. mm. Hg.) in order to avoid decomposition. Many investigators have advocated boiler pressures of below .5–1 cm. of pumping fluid as a maximum if decomposition is to be avoided even with backing pressures as low as .02 mm. Hg.

Higher vapor pressure pumping fluids can be used efficiently against backing pressures substantially in excess of .2 mm. such as at .5–10 mm. if the organic vapors are generated in the boiler under high vapor pressure. Sufficient kinetic energy is thus imparted to the pump vapors to enable working against the higher backing pressures. Although the increased boiler pressures slightly decrease the degree of vacuum obtainable it has been found that at the higher boiler pressures, the speed of the pump is not only unimpaired, but is actually substantially increased. This is possibly due to the fact that the pump is handling gases which are at a pressure intermediate between that at which the Knudsen type of flow and the Poiseuille type of flow takes place.

While such a pump will not give as low a pressure as the best organic condensation pumps the benefits of both may be utilized if desired by connecting them in series. Thus a condensation pump using octyl phthalate will give a vacuum as high as 10^{-7} mm., but will operate with efficiency and without decomposition only against backing pressures substantially below .1 mm. However, if a booster pump employing a high vapor pressure of pumping fluid is interposed between the octyl phthalate pump and a backing pump, efficient evacuation to 10^{-7} mm. or below will be obtained with considerably higher backing pressures. Backing pumps having high capacity such as ordinary mechanical pumps can thus be employed with organic pump fluids where they were previously unsuitable. Also high velocity multi-stage steam ejectors which are known to be very efficient can be used for the same purpose.

Organic fluids which may be employed as operating fluids in the booster pump should be capable of withstanding vaporization under the relatively high boiler pressures without substantial decomposition. Compounds having a vapor pressure considerably higher than that of the pump fluid in the fine pump are subject to less decomposition and can be vaporized at higher pressures and maintained at high boiler pressures without substantial loss. It has been found that pump fluids having a vapor pressure the same as or higher than di-amyl phthalate may be used in the booster pump. Such compounds can be vaporized without decomposition at such a temperature that their vapor pressures are sufficiently high to operate against high backing pressures.

Since the backing pump pressure must be less than the boiler pressure of the booster pump, it is apparent that the selection of the particular booster pump liquid will depend upon the backing pressure against which it is to operate. As a general rule it may be stated that the lower the vapor pressure of pump fluid the lower must be the backing pressure. Amyl phthalate represents the lowest vapor pressure pump fluid which may be employed, and it therefore requires a low backing pressure of below 5 mm. and preferably of the order of about .4 to 1 mm. for efficient pumping. Although backing pressures within this range can be efficiently obtained with steam ejectors or high capacity single mechanical pumps I prefer to employ fluids which have a higher vapor pressure than amyl phthalate in order to avoid all possibility of decomposition at these or higher backing pressures. Examples of such fluids are petroleum distillates having a vapor pressure not greater than .1 mm. at 20° C. or less than .1 mm. at 160° C., dimethyl, ethyl, propyl, and butyl phthalates, diglycerol tetrapropionate, tripropionin, tributyrin, triamylin, amyl naphthalene, dimethyl and ethyl sebacates, squalene and the hydrocarbon obtained by the molecular distillation of a fish oil disclosed in U. S. application #84,870 filed June 12, 1936, by J. G. Baxter. Such substances will operate against higher backing pressures and will withstand decomposition at the higher boiler pressures and temperatures. Any other organic fluid can be used which has the required vapor pressure and stability.

In order to retain a pumping fluid of constant composition and also avoid loss of pumping fluid resulting in a dry boiler, it is a distinct advantage to place a fractionating condenser between the booster pump and the backing pump. In this way all useful components can be condensed and returned to the boiler of the pump and light ends can be segregated and either allowed to be drawn into the backing pump or retained in the fractionation device. A separate boiler can be used for this purpose or the boiler of the pump itself can be made to supply the vapors to the fractionator. Where several pumps are used in series, each being provided with a different vapor pressure fluid, contamination of one pump fluid with another can be prevented by providing each pump with a fractionating device. The volatiles will thus be segregated and thrust back to the higher vapor pressure pump from which they came. For a more detailed description of the construction and mode of operation of such devices, reference is made to my co-pending application #27,652 filed June 21, 1935.

With the foregoing explanation the principles of my invention will be more fully understood by reference to the accompanying drawings in which:

Fig. 1 illustrates in diagrammatic form an elevation in section of an assembly of a series of condensation and ejector pumps embodying the principles of my invention and:

Fig. 2 illustrates a sectional elevation of a preferred form of booster or condensation pump provided with a fractionating device.

Referring to Fig. 1 reference numeral 1 designates a curved conduit connected at one end to a chamber to be evacuated and at the other end to conduit 2 which communicates with the intake chamber or low vacuum side of booster pump B. Reference character A designates a condensation pump mounted upon the bottom inside wall at the lowest portion of the curve of conduit 1, having an opening 3 in the side thereof and a flared jet 4, and numeral 16 designates

the boiler and body of pump fluid. Pump B comprises a boiler 5, a vapor conduit 6 communicating with expansion nozzle 7 having a restricted portion 8. Surrounding the nozzle is a chamber 9 communicating with conduit 2 and with condensing tube 10 provided with external cooling jacket 11 through which cooling fluid is circulated by means of conduits 12 and 13. Pump 14 serves to return pumping fluid condensed in 10 to boiler 5. Conduit 15 connects the lower portion of condensing member 10 with the intake or low pressure side of high velocity steam ejector pump C the high pressure side of which is connected to the intake chamber of steam ejector D by conduit 20. Steam ejector pump E withdraws gases from the high pressure side of pump D through conduit 21 and ejects them through exhaust 22 which is at atmospheric pressure.

Super-heated steam for operation of ejector pumps C, D and E and for heating condensation pump B is admitted into the system through valve 22 and conduit 23. Pumping fluid in boiler 16 of pump A is vaporized by an external packed heating jacket 24 provided with electrical resistance coil 25. Boiler 5 of pump B is heated by steam passed through a spiral-shaped conduit 28 connected to 23 by means of conduit 29 provided with valve 30. Exhaust steam is withdrawn through conduit 31 and preferably delivered to a suitable heat regenerator. Since the details of construction of multi-stage high velocity steam ejectors do not form a part of this invention and are well known in the art, only a general illustration of their construction and mode of operation have been given.

Referring to Fig. 2 reference numeral 50 designates the cylindrical casing of a booster pump which is closed at the base by integral plate 51 and at the top by plate 52 which makes a gas tight joint with flange 53 by interposing a gasket or suitable sealing compound. The base of cylinder 50 is cut out to form supporting legs 54 and openings 55 are provided for exit of combustion gases from burner 56 which is supplied with combustible fuel through conduit 57. A gas intake conduit 58 is located at the upper end of 50 and is connected to a condensation pump or the chamber to be evacuated. A concentric cylinder 59 is located inside casing 50 and is maintained in the position shown by the flange 60 which is welded to the internal walls of the casing. Numeral 61 designates a body of pumping fluid which is located in the boiler 62 and numeral 63 a conduit connecting the space above flange 60 with the boiler, and terminating below the surface of fluid 61. A rod 64 is passed through a centrally located packed gland 65 and is attached at the lower end to a three legged centering spider 70, the legs of which touch the inside walls of 59 but are free to move in a vertical direction. A cylindrical false top 71 is fastened to rod 64 by means of integral plate 72 in such a manner that it is concentric with 50 and 59. The periphery of 71 is flared as shown to form an annular jet 74 similar to those of the well-known umbrella type of condensation pump. Plate 72 is provided with a plurality of openings 73 as shown. A tube 75 is centered around rod 64 and is threaded at the top and mounted in gland 76 in the same manner as rod 64. The lower end of 75 is attached to flange 77 which is flared downward at the periphery to form an annular jet 78. The tops of rod 64 and tube 75 are provided with knurled knobs 79 and 80 so that they can be turned to adjust the openings of jets 74 and 78. To prevent entry of gas glands 65 and 76 are provided with reservoirs 81 and 82 which are filled with a low vapor pressure sealing liquid.

A condensing element 83 is located on the outside of 50 and integral therewith, cooling fluid being circulated through the conduits shown. A conduit 84 slanted in an upward direction and terminating in a substantially vertical portion 85 is connected to the annular space formed between cylindrical elements 50 and 59. The upper portion of 85 is closed by plate 86 to exclude entry of air. Conduit 87 leads to a backing pump such as a steam ejector or mechanical pump. The central portion of 85 is provided with an external cooling element 88, cooling fluid being circulated through conduits 89 and 90. A plurality of gutter-like fractionating trays 91 are located upon the internal walls of conduit 85 and serve to fractionate pumping vapors as they are condensed. The top plate is provided with a withdrawal conduit 92 for removal of the lightest ends. Most of the vapors are supplied to elements 91 by a boiler 93 connected by the top plate 94 to conduit 95 the end of which terminates near the lowest fractionating tray 91. The base of boiler 93 is shaped similar to that of 62 and is provided with a burner 96. The base plate 97 is at a slightly lower level than that of plate 51 so that fluid 61 flows at a slow rate through conduit 98 provided with screen 101 and into 99. Removal or addition of fluid is accomplished by introduction through conduit 100. A conduit 110 communicates at its uper end with the lowest tray 91 and at its lower end with conduit 84. It is provided with a drop counter 111 in which is located a glass window 112. A glass tube 115 connects conduit 58 with the lower and upper portions of boiler 62 by branched conduits 116 and 117. This serves as a manometer indicating the pressure in the boiler as well as the height of the pump liquid. A series of baffles 118 are mounted upon shaft 119 which is centrally located in conduit 85 by spiders 120 and 121. These baffles prevent the withdrawal of pump vapors into the backing pump when a rapid stream of gases is removed.

In operation employing the apparatus of Fig. 1 conduit 1 is connected to the chamber to be evacuated. Superheated steam for instance at a temperature of about 170° C. is admitted through valve 22 and the system evacuated by steam ejectors C, D and E. Valve 30 is then opened and the pump fluid in B heated to vaporizing temperature under a boiler pressure considerably in excess of .3 mm. Hg. Vapors pass through conduit 6 and restriction 8 and expand in a relatively high velocity jet into condensing chamber 10. Gases arriving through conduit 2 are entrained in the jet and forced into conduit 15 from which they are removed by the steam ejector pumps. Liquid condensed in 10 flows to pump 14 and is returned to boiler 5. Pump B when filled with di methyl phthalate and heated sufficiently to give a boiler pressure of 5–25 cm. of pump fluid will rapidly produce a vacuum of below .05 mm. against a backing pressure of between .5 and 10 mm. Hg. After pump B has been in operation and reduced the pressure of the system to substantially below .1 mm., pump A is started by electrical heating unit 25. A low vapor pressure pump fluid such as di-octyl phthalate or "Apieson" oil is employed in pump A. Vapors from pump A flow through jet 4 and are condensed on walls of conduit 1, condensed

liquid flows down the bottom of conduit 4 and again enters the boiler 16 through opening 3. To prevent loss of vapors through this opening the pump fluid level should be above the highest point of 3. The rate of vaporization and temperature of pump fluid in boiler 5 is controlled by the amount of steam flowing through valve 30. Final pressures of about 10^{-7} mm. and below can be efficiently produced by this system.

In operating the apparatus of Fig. 2 the boilers 62 and 99 are filled, to about the level indicated, with a suitable booster pump fluid such as butyl phthalate. The conduit 58 is connected to the chamber to be evacuated, or to the high pressure side of a condensation pump if it is to be used in series, and conduit 87 is connected to a backing pump which is put into operation. After the pressure in the system has been reduced by the backing pump, burner 56 is started and the butyl phthalate vaporized under a boiler pressure of 4 to 12 cm. head of butyl phthalate. The vapors travel up through 59, part of them enter the openings 73 and flow in a reversed direction through annular jet 78. The balance of the vapors flow through jet 74. The vapors are thus expelled as a vigorous double jet into the passage formed between cylinders 50 and 59. Gases flowing through conduit 58 are entrained in the jet and forced downward into conduit 84 and are withdrawn by the backing pump connected to 87. The heavier vapors are condensed by contact with the cooled walls in the vicinity of cooler 83 and flow down the walls of 50 onto flange 60 and are returned to the boiler by conduit 63. The optimum jet openings are obtained by varying the distances by means of adjustment knobs 79 and 80.

During the pumping action burner 96 is started and the pump fluid in 99 vaporized. The vapors travel up through conduit 95 and are condensed in 85. Due to the fractionation effect light ends condense in the top rings and can be withdrawn through conduit 92. Heavier components are fractionated and those free of light materials flow from the lowest ring 91 into conduit 110 and back into the boiler of 62 via conduit 63. During the functioning of boiler 99 fresh fluid flows from 62 into 99 by way of conduit 98. The number of rings 91 and the degree of cooling at 88 can be varied greatly to separate any desired range of volatile materials. The pump fluid is therefore easily maintained at any predetermined composition. A particular advantage of the fractionation construction is that loss of pump fluid resulting in a dry boiler and destruction of the vacuum is prevented. Pumps of this type can be operated continuously for months which would be impossible with old types of construction. Pressure in boiler 62 is adjusted by varying the openings of the jets and the amount of heat supplied.

Many changes can be made in the apparatus illustrated and in its mode of operation without departing from the spirit or scope of my invention. It is possible, for instance, to place a trap containing a hygroscopic agent such as calcium chloride or sodium hydroxide, between the steam ejector and the booster pump, thus avoiding any possibility of water vapor traveling into the latter. Instead of using one or two booster pumps any number can be used in series to give a gradually increasing degree of vacuum. Obviously if the booster pump gives a vacuum sufficiently high for the particular purpose it need not be used in combination with a high vacuum condensation pump. The use of fractionating devices is preferred, but the pumps will work without them. Baffles 118 of Fig. 2 can be eliminated if the volume of gas removed is insufficient to carry material amounts of pump fluid vapor into the backing pump.

The essential distinction between booster pumps as described and condensation pumps heretofore known is in the pump fluid employed, the method of operating the pump and the use of fractionating expedients. The construction must be changed somewhat from that of known pumps to enable higher pressures to be developed in the boiler and to prevent back passage of vapors into the high vacuum side, but they are otherwise much the same in detail of construction and the booster pumps can be made in a variety of forms now common to condensation pumps such as vertical or inverted jet, mushroom and multiple-jet mushroom types. In order to generate the pump vapors a restricted jet or a divergent or expansion type nozzle or jet with restricted throat is used thus preventing the generated vapors from escaping as fast as formed. The vapors are thus generated under higher pressure and escape through the expansion nozzle as a vigorous vapor stream. Due to the high velocity of the jet, proper selection of the expansion ratio and the use of small clearance, the back passage of vapors into the chamber to be evacuated or into the condensation pump is prevented. These details of construction, as is well known, vary according to the size of the pump, the particular fluid used, boiler pressure etc. and cannot therefore be empirically defined. However the construction in each particular case will be obvious to those skilled in the art of nozzle and jet pump design.

As indicated above amyl phthalate having a vapor pressure of about 2×10^{-6} mm. at 20° C. represents the lowest vapor pressure which a booster pump fluid may have if decomposition is to be substantially avoided. The upper vapor pressure limit depends entirely upon what vacuum it is desired to produce. If a vacuum of .05 is required, a pump fluid having a vapor pressure at the temperature of the pump condenser of .05 mm. or below is required. Fluids having the required stability and vapor pressures of between about .05 and about 2×10^{-6} mm. at room are satisfactory, those having a vapor pressure of between about .01 and 2×10^{-5} mm. being most generally useful. The boiler pressures used can be so high that they approach the decomposition point of the liquid used. For liquids having a vapor pressure in the higher portion of the above range, boiler pressures of up to 20 mm. Hg. are usually most satisfactory. Boiler pressures of up to 15 mm. such as between 10 and 2.5 mm. Hg. are generally used, the lower values being best when using liquids having the lower vapor pressures. The booster pumps will operate efficiently against backing pressures substantially above .3 mm. such as .5–10 mm. These pressures can easily be obtained with ordinary mechanical pumps or steam ejectors. Backing pressures of .5–2 mm. are desirable with the lower vapor pressure liquids especially when used with large pumps designed for high capacity, but higher pressures can be used with efficiency.

While I prefer to use steam or other heated vapor as a means of heating the lighter or higher vapor pressure pump fluids it is apparent that other heating means such as electricity can be used. Vapors such as steam have the distinct

advantage that quick cooling of the boiler is obtained when the heating fluid is shut off. This factor is of considerable importance in preventing destruction of the pumping fluid when the vacuum is broken due to accident or trouble with backing pumps. Also overheating will be avoided and rapid starting therefore possible due to the fact that no temperature above that of the steam can be applied to the pump fluid.

Although I have employed the terms, high pressure, relatively high boiler pressure, etc., it is to be understood that the invention relates to vacuum and especially high vacuum production and that these expressions have the meaning which would generally be given them in this art. In the claims all pressures are in millimeters of mercury.

What I claim is:

1. Apparatus for exhausting closed receptacles comprising in combination, a condensation pump filled with an organic pump fluid having a vapor pressure between .05 and 2×10^{-6} mm. at 20° C. and a high velocity steam ejector connected on the low vacuum side.

2. Apparatus for exhausting closed receptacles comprising in combination a condensation pump the boiler of which is filled with an organic pump fluid having a vapor pressure between .01 and 2×10^{-5} mm. at 20° C. and a high velocity steam ejector connected on the low vacuum side of the pump.

3. Pumping apparatus for exhausting a closed receptacle comprising in combination a condensation pump the boiler of which is filled with an organic pump fluid having a vapor pressure between .05 and 2×10^{-6} mm. at 20° C. and a high velocity steam ejector connected to the low vacuum side of the condensation pump the condensation pump being provided with fractionating means for separating the volatile constituents from the pump fluid.

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