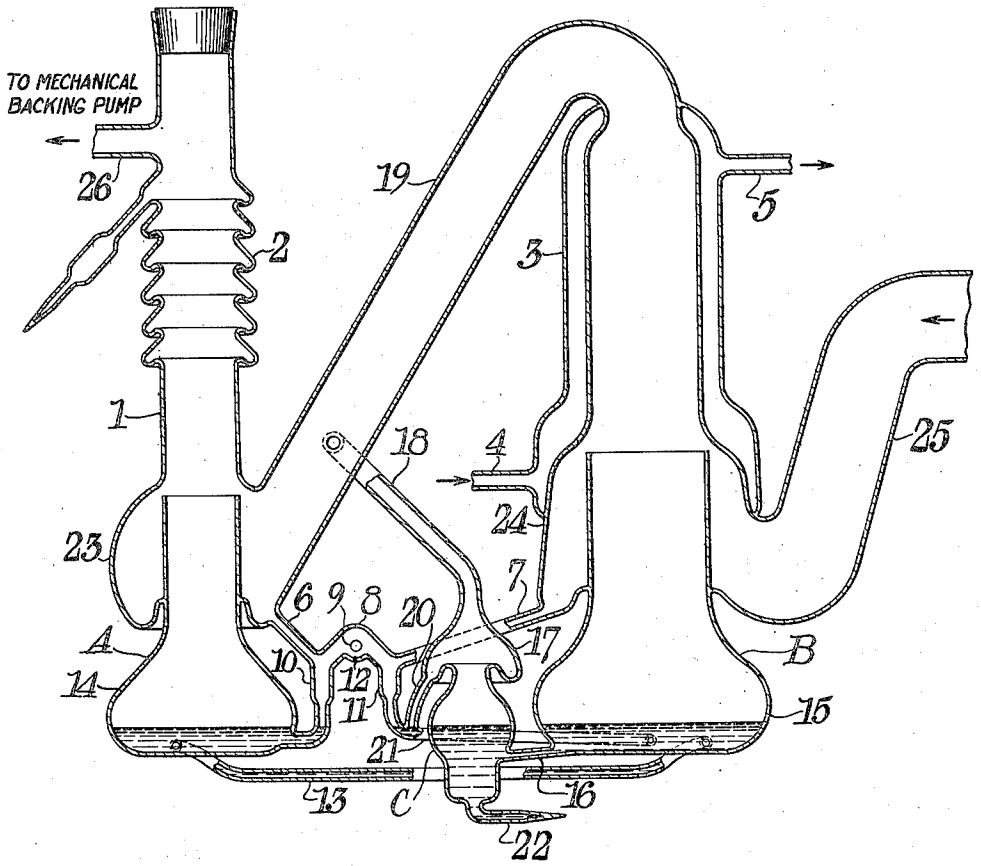
May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 1

Fig. 1.



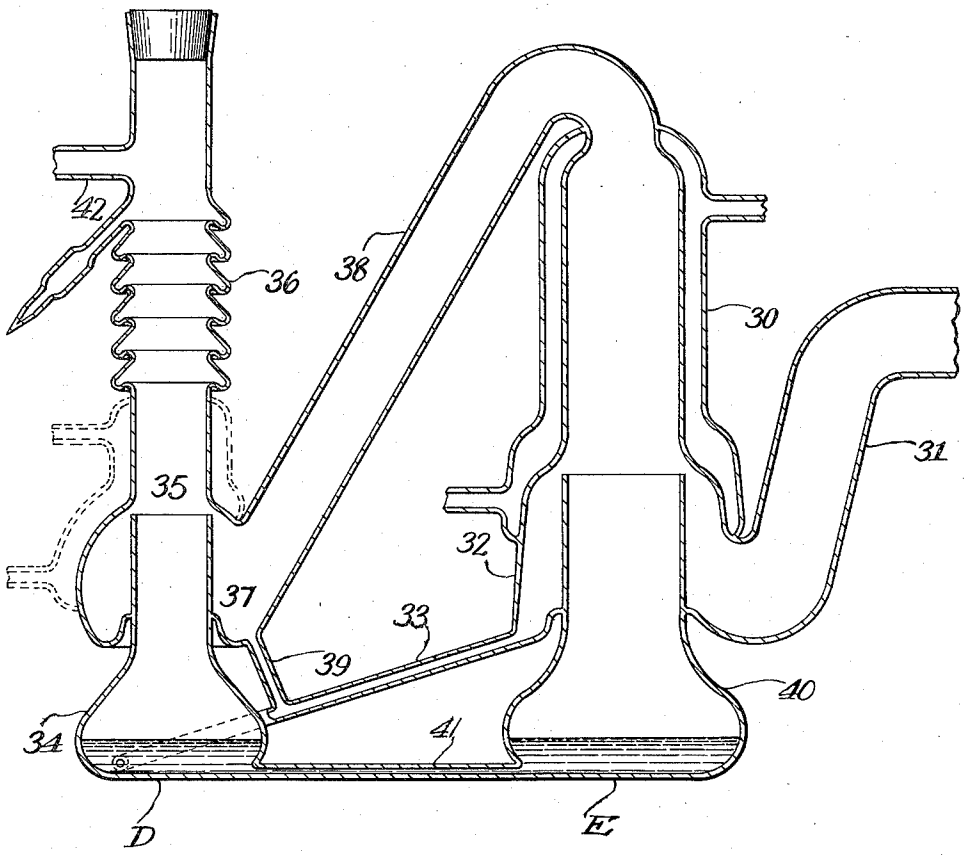
INVENTOR:
Kenneth C. D. Hickman
BY Newton M. Perrins
Henry S. Boynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 2

Fig. 2.



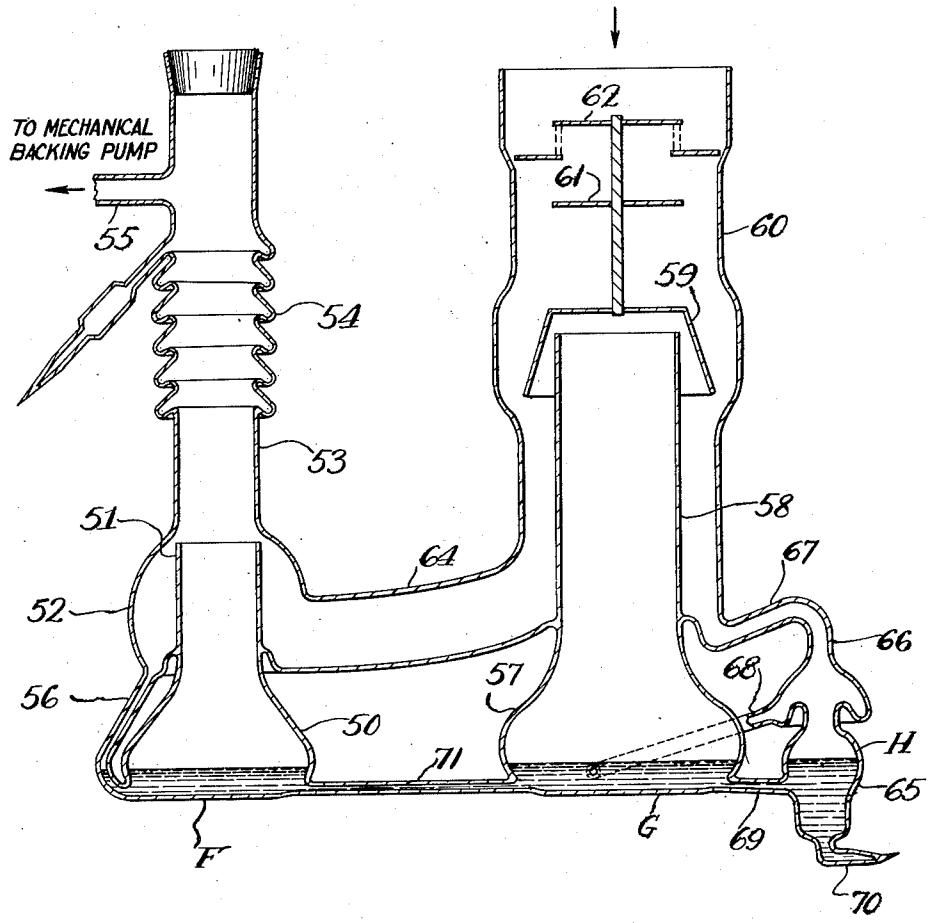
INVENTOR:
Kenneth C. D. Hickman,
BY Newton M. Perrins
Henry S. Boynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 3

Fig. 3.



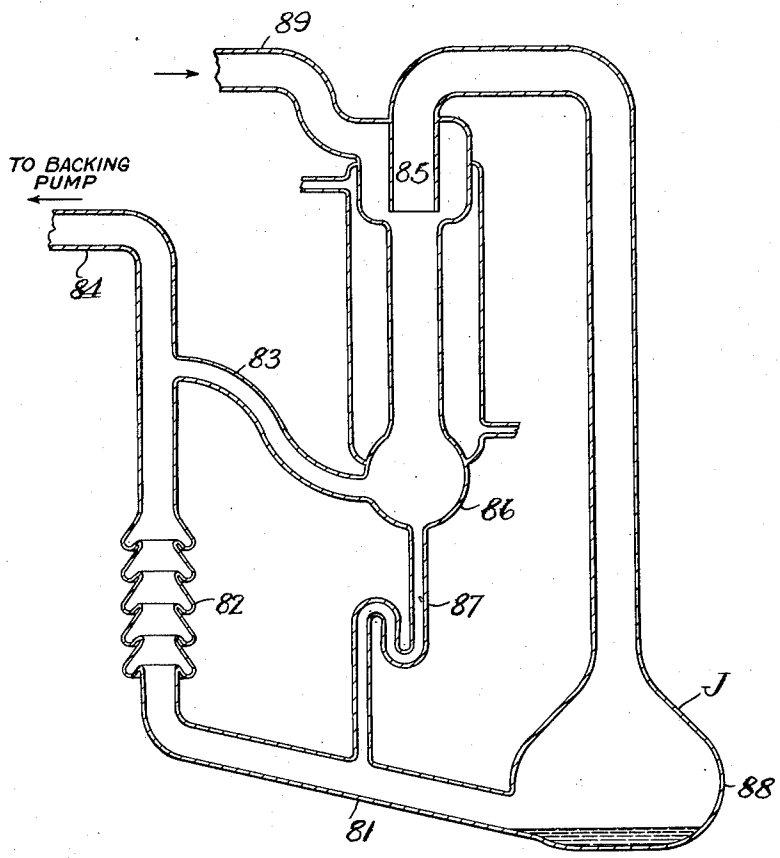
INVENTOR:
Kenneth C.D. Hickman,
BY Newton M. Pierce
Henry S. Boynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 4

Fig. 4.



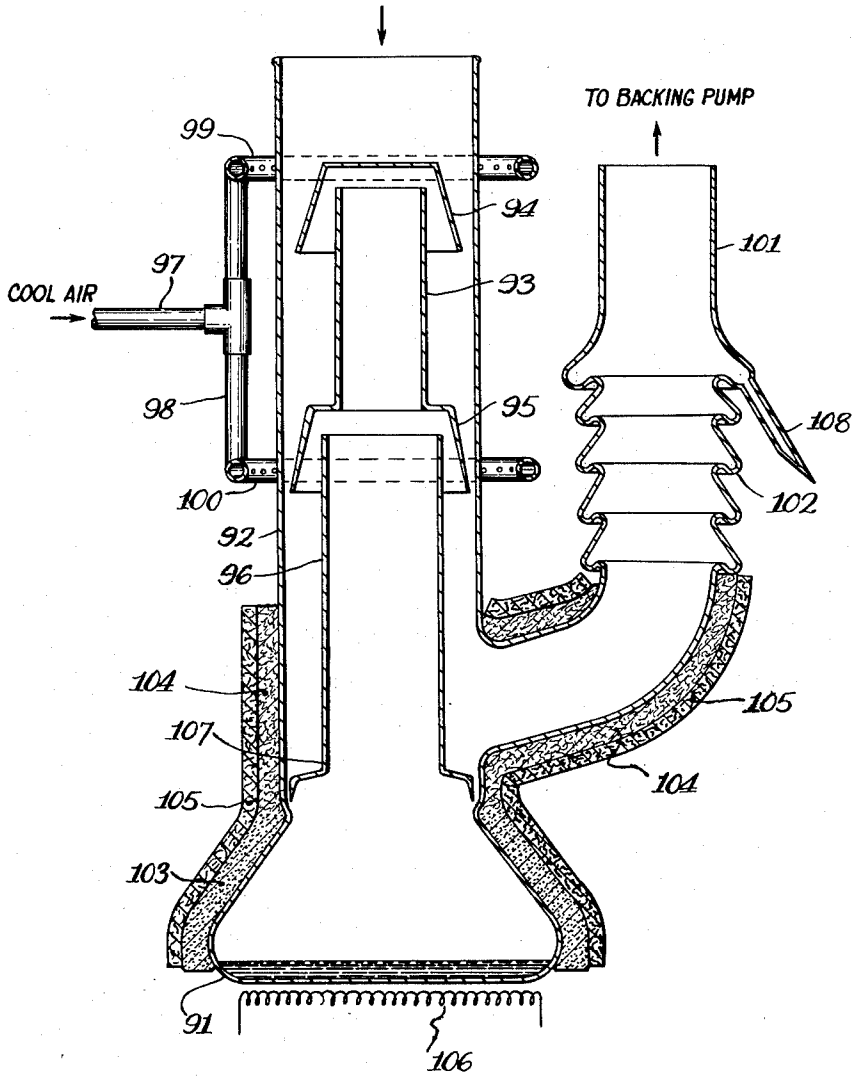
INVENTOR:
Kenneth C. D. Hickman,
BY Newton W. Perrins
Henry S. Boynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 5

Fig. 5.



INVENTOR:
Kenneth C. D. Hickman,
BY Newton M. Perkins
Henry S. Boynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 6

Fig. 6.

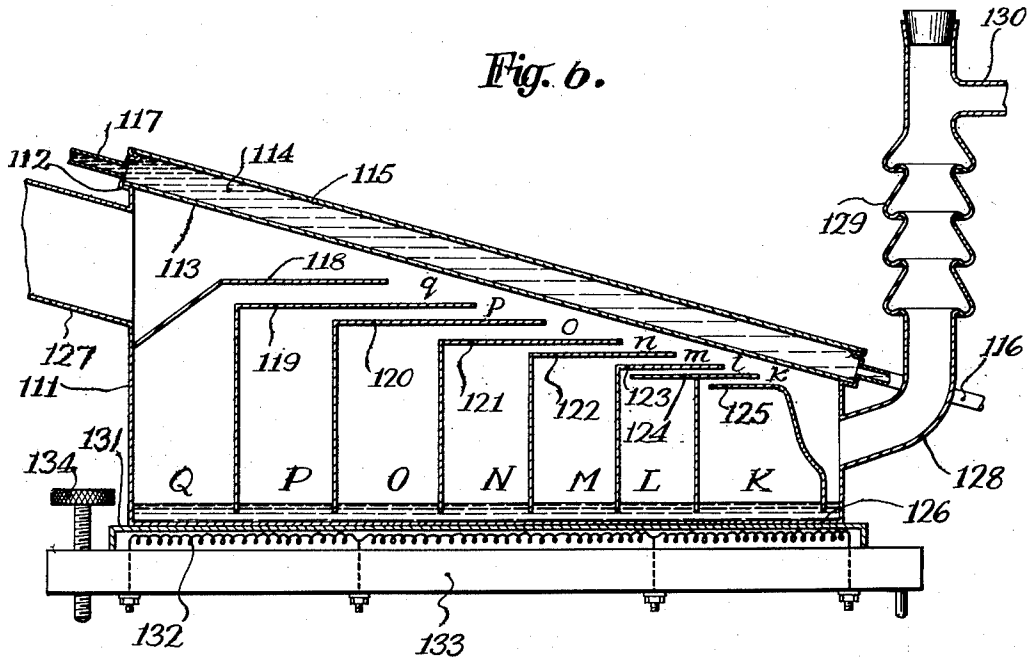
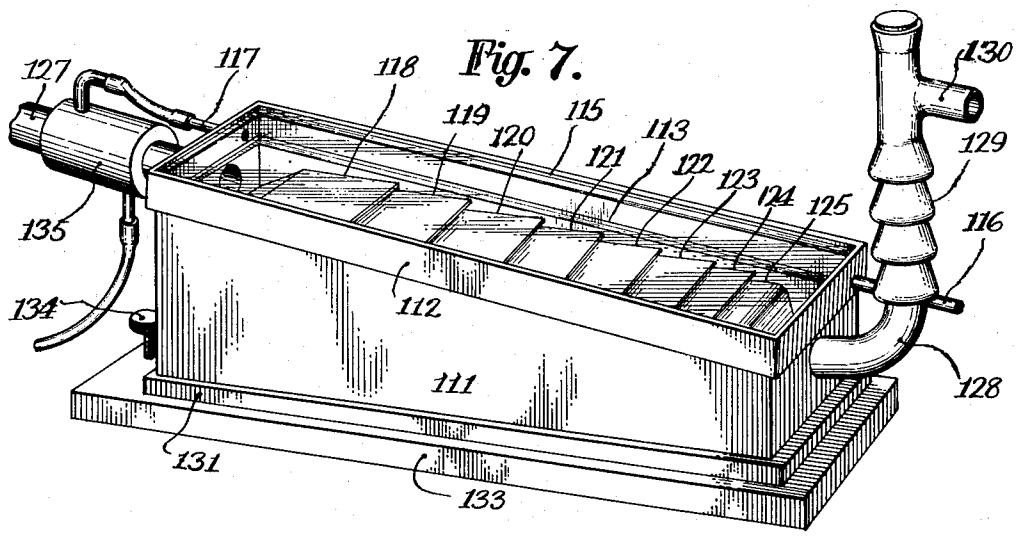


Fig. 7.



INVENTOR:
Kenneth C.D. Hickman,
BY Newton W. Perrin
Henry S. Baynton
ATTORNEYS.

May 18, 1937. K. C. D. HICKMAN 2,080,421

VACUUM PUMP

Filed June 21, 1935 7 Sheets-Sheet 7

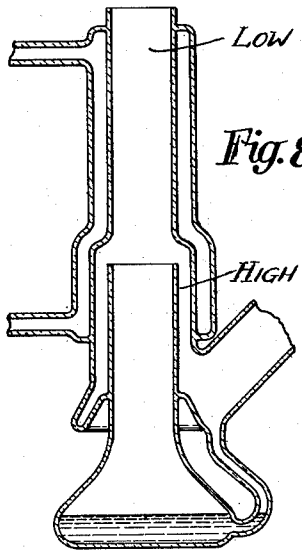


Fig. 8.

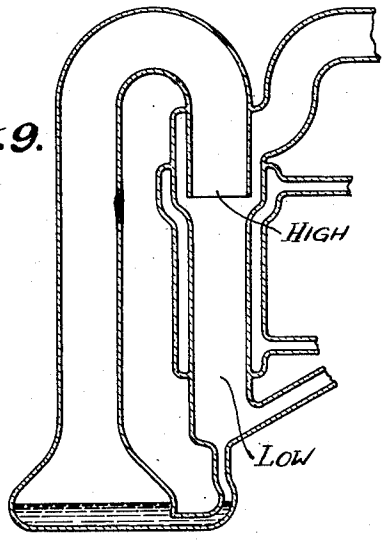


Fig. 9.

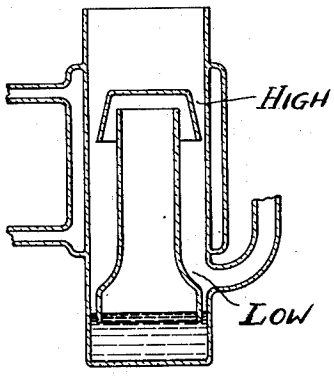


Fig. 10.

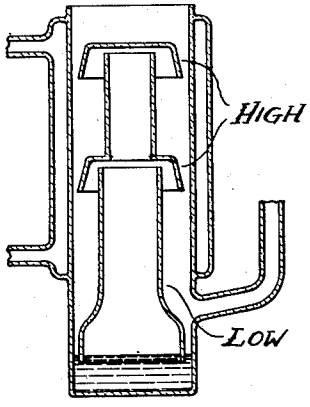


Fig. 11.

INVENTOR:
Kenneth C.D. Hickman,
BY Newton M. Parris
Henry S. Boynton
ATTORNEYS.

Patented May 18, 1937 2,080,421

UNITED STATES PATENT OFFICE

2,080,421

VACUUM PUMP

Kenneth C. D. Hickman, Rochester, N. Y., assignor, by mesne assignments, to Eastman Kodak Company, Jersey City, N. J., a corporation of New Jersey

Application June 21, 1935, Serial No. 27,652

18 Claims. (Cl. 230—101)

This invention relates to a method of and apparatus for producing high vacua, and more particularly to diffusion pumps especially adapted for employing organic working fluids containing, or giving off under the conditions of operation, larger or smaller amounts of relatively volatile substances.

The diffusion method of obtaining high vacua has been known for many years. The pioneer work of Gaede about 1914 was extended by Langmuir in the development of the vertical jet diffusion pump covered by U. S. Patent 1,393,350, and the single inverted jet or mushroom pump covered by Patent 1,320,874. A multiple jet modification of the mushroom pump was subsequently developed by Crawford and is described in Patent 1,367,865. For convenience of reference and comparison, these conventional pump structures are illustrated in the last four figures of the drawings and will be adverted to in connection with the detailed description of the present invention appearing hereinafter.

The use of mercury as the working fluid, although presenting certain disadvantages referred to in my prior Patent 1,857,506, is a comparatively simple operation and fairly consistent results have been obtained with conventional pumps. However, with the introduction in recent years of new pumping fluids such as the phthalate esters described in my U. S. Patent 1,857,508 and certain of the hydrocarbon oils suggested by C. R. Burch (Nature, 1928, 122, 729) and sold under the trade names Apiezon oils, much of the original simplicity has disappeared and uniform results have been found to be practically unattainable.

It has been assumed since Gaede's work (Annalen der Physik 1915, 46, 357) that the lowest pressure obtainable by a vapor pump should correspond with the vapor pressure of the working fluid in the coldest part of the high vacuum side of the system. With mercury, on a summer day and in the absence of a special cooling trap, this proves to be about 5×10^{-3} mm., agreeing well with theory. With various organic working fluids, however, such as the Apiezon oils and n-dibutyl phthalate having real vapor pressures at room temperature ranging from 10^{-6} to 10^{-8} mm. and 2×10^{-4} mm., respectively, corresponding vacua have seldom, if ever, been attained in actual practice, many of the reported results of workers in high vacuum technique being open to serious suspicion because of the use of obviously defective methods of measuring the pressures of mixed gases prevailing in such systems.

It has been suggested by research workers using the Apiezon type of fluids, which are mixtures of substances of varying volatility, that the lack of uniformity in the results theretofore obtained and the fact that vacua corresponding to their effective vapor pressures were unattainable, might be due to lack of homogeneity of these oils in comparison with mercury. I have examined this suggestion and found that, not only is it true for mixed oils, but that even the most carefully prepared individual organic fluid is likely to contain traces of both lighter as well as heavier constituents. I have further found that under the conditions of operation organic fluids are thermally decomposed to a certain extent giving off gases and volatile compounds. Experience with vapor pumps of conventional design has demonstrated that these small amounts of impurities in a so-called "pure" working fluid and the "light ends" in a fluid of the Apiezon type collect in the wrong places in the pump, the volatiles which spoil a vacuum diffusing into the high vacuum side, while the less volatile materials which could improve a vacuum remain inactively in the boiler.

The adverse effects of small amounts of volatiles and other impurities can be conveniently illustrated with reference to *n*-dibutyl phthalate, although the higher phthalates and other organic fluids suffer similar disadvantages. Normal butyl phthalate, even though prepared with extreme care, contains traces of isomeric butyl phthalates boiling about 20° C. lower than the main portion of fluid; of water boiling 250° lower; and of air boiling some 530° lower. Under the conditions of operation, thermal decomposition may give rise to butyl alcohol, butylene and carbon dioxide, all boiling some hundreds of degrees lower than the original phthalate. After prolonged use in a pump, less volatile polymers and tarry residues are formed and collect at the evaporating surface, raising both the real and apparent boiling points of the fluids (by repressing evaporation). The temperature rises and decomposition proceeds in a vicious circle. As indicated, pump oils other than butyl phthalate are not immune to these defects, the foreign bodies resulting from the use of such liquids differing merely in kind, but not in effect.

This condition may be conventionalized by assuming that a pump fluid consists of a number of components designated alphabetically from A to Z. The distribution of quantities in a "pure"

homogeneous liquid such as a phthalate ester is roughly $A+B+(C——Y)+Z$ and in an oil of the Apiezon type: $A+B+(C+D$, etc., to $W+X+Y)+Z$, where A comprises the permanent gases and water vapor, B the alcohols, phthalic anhydride or other "light ends," and Z the phlegmatic residues. In the first example (butyl phthalate), the portion C——Y is homogeneous and has a boiling point of 104° C. at 0.1 mm. of mercury. In the second example, the various constituents boil between 130° C. and 170° C. The harmful components are A, B, and Z, in each case, though Z is of secondary importance and matters only if it contributes materials to A or B.

Consider what happens when an oil is boiled in a pump such as one of the conventional types shown diagrammatically in Figures 8 to 11. In a short time the constituents A and B, which spoil a high vacuum, are emitted from the jet and have an opportunity of diffusing to the point of highest vacuum indicated in Figures 8 to 11 by the word High (the region of low vacuum being indicated by the word Low). Not all the volatiles will diffuse from the main stream of vapor at each opportunity; and some of the volatiles that have already accumulated in the high vacuum leads will rediffuse back into the jet. There will be a dynamic equilibrium between the concentration of volatiles in the vapor issuing from the jet and those remaining in the high vacuum leads. It is manifest that when an opportunity is given for elimination of the volatiles at a point other than on the high vacuum side, the equilibrium will be disturbed and volatiles will partition themselves between the high vacuum leads, the boiler and the other reservoir for volatiles. In the inverted types of pump of Figs. 9, 10 and 11 there is no low vacuum reservoir and any volatiles liberated under high vacuum from the hot jet will redissolve more or less completely in the cold condensate at the low vacuum part of the pump. They will be returned to the boiler to be started on their cycle again, together with any additional volatiles which have accumulated during the boiling operation. On the other hand, the vertical type of pump, shown in Fig. 8, has a natural alternative reservoir for the volatiles.

Often criticized, because the warm condensate, falling backwards, limits the pumping speed, this type of pump continually causes the volatiles to be thrust away from the boiler, the chimney forming a miniature fractionating column at the top of which the unwanted constituents collect. In considering the plausibility of this statement, it is well to remember that continuous rejection is possible because the condensed volume of impurity is often less than one drop. The favorite double-stage mushroom pump (Figure 11) is a particularly gross offender with mixed oil fillings. The long, circular tube separates the C—X portion of fluid, using C (the lower boiling, higher vapor pressure component) at the high vacuum jet and X (the higher boiling, lower vapor pressure component) at the low vacuum side of the system in the wrong sequence but, since these pumps are designed to work against a higher backing pressure than the single stage variety, the A—B constituents are more thoroughly redissolved on the return journey of the fluid to the boiler.

A suggested method of eliminating the harmful effects of diffusion of the lower boiling components of the pump fluid into the high vacuum side is to cool the condensing tube slightly below room temperature and to arrange the location of the fore (low) vacuum pipe and the heat conductance of the various parts of the pump so that the fore vacuum pipe runs slightly warm.

This method cannot be considered desirable, for extra cooling applied to the high vacuum side will attract in greater quantity the very volatiles it is hoped to reject. The application of cold at this point is equivalent to an admission that the vapor pressure of the working fluid is too high for its intended purpose. Similarly, allowing the fore vacuum side of the pump to run warm confronts the operator with the question How warm? If not warm enough, the class B volatiles are not eliminated. If too warm, the contents of the pump may be distilled away, leaving it empty.

It might be supposed that mixed pumping fluids should be abandoned in favor of substances which have been extremely carefully purified. Such a supposition is erroneous, however, for even the purest liquids alter during use, acquiring impurities, either spontaneously or from interaction with the gases or vapors passing through the pump. It is more reasonable to conclude that means should be provided whereby organic liquids such as the phthalate esters and the Apiezon oils and other mixed fluids may be used in spite of their inherent defects.

The present invention, accordingly, has as its object to overcome the above-mentioned difficulties. A further object is to provide a mechanism for continuously purging and purifying pumping fluids during use. A still further object is to provide a pumping mechanism wherein the volatile portions of the working fluids are continuously thrust towards the low vacuum or high pressure region where they can exert no harmful effect on the ultimate vacuum. A further object is to provide a pumping mechanism which shall create a vacuum equal to the vapor pressure of the pure working fluid at the temperature obtaining in the high vacuum side of the pump. A further object is to provide an improved type of diffusion pump which is adapted to employ mixed organic and other types of multiple-component working fluids with a high degree of efficiency and by means of which hitherto unattainable vacua can be produced. Other objects will appear hereinafter.

Contrary to what would be expected from the results hitherto attained, and at variance with the suggestions of workers in high vacuum technique, I have found that the above objects can be accomplished by providing a technique whereby the various components of the working fluid will be separated and caused to perform their respective pumping functions in proper order, that is, a technique in which provision is made for the above-mentioned continuous fractionation of volatiles and purging of the pumping fluid of tarry residues and other relatively non-volatile materials which, either have an undesirable effect on, or contribute nothing to, the pumping efficiency. In order to carry out such a method, I have developed an improved type of diffusion pump, the various modifications of which are shown in the accompanying drawings in which:

Figure 1 is an elevation in section of an experimental multiple vertical jet type of diffusion pump designed to illustrate the improvement obtained by operating a vacuum pump in accordance with the present invention.

Figure 2 is an elevation in section of a pump suitable for routine use in the laboratory, modeled after the experimental design of Figure 1.

Figure 3 is a sectional elevational view of a simple inverted jet vacuum pump modified in accordance with my invention.

Figure 4 is a sectional elevation of a pumping apparatus in accordance with my invention employing both inverted and vertical jet pumping units.

Figure 5 is a sectional elevational view of a modified multiple inverted jet pump.

Figure 6 is a sectional elevational view of a multi-jet cascade pump in which the jets are arranged horizontally.

Figure 7 is a perspective view of a pump structure similar to that illustrated in Figure 6.

Figures 8, 9, 10 and 11 are diagrammatic elevations in section of conventional diffusion pumps of the vertical jet, inverted jet, mushroom and multiple mushroom types respectively.

In the following examples and description I have set forth several of the preferred embodiments of my invention but they are included merely for purposes of illustration and not as a limitation thereof.

My invention may first be conveniently described by reference to a pumping experiment in which butyl phthalate is employed as the operating fluid. Accepting the description of this material as a liquid containing three components namely, (A—B) (C——Y) (Z), as described above, it is seen that a pump having at least three compartments is required which shall afford small storage space for the (A—B) and the (Z) constituents and a larger space for the main bulk of the fluid. This may be provided in accordance with my invention by mounting two simple glass pumps in tandem as illustrated by the experimental device of Figure 1, the most consistent results being obtained with a double vertical jet pump. A is the fore pump with a short receiving jet 1 (which need not be water-jacketed) terminating in a series of small re-entrant bulbs 2 designed to retain a few cubic centimeters of fluid. The fine pump B on the high vacuum side is larger and is equipped with a water jacket 3 supplied with inlet 4 and outlet 5 for the cooling water. When the system is operating, the condensates from A and B travel by conduits 6 and 7, respectively, to a junction 8 containing a steel ball 9 (in the equipment shown a $\frac{3}{16}$'' diameter ball bearing is suitable) which can be moved by an external magnet either into pocket 10, comprising a continuation of conduit 6, or into pocket 11, a continuation of conduit 7, or allowed to remain inactive in the center depression 12 of junction 8.

A conduit 13 connects the two boilers 14 and 15, while a third conduit 16 allows fluid to pass from B to a small evaporator C. C is provided with a re-entrant bulb 17 for collecting condensate and this bulb communicates with conduit 18 which passes gas or vapor from boiler C into conduit 19 connecting pumps A and B. Re-entrant bulb 17 of boiler C communicates by means of conduit 20 with a conduit 21 connecting pocket 11 with boiler 15 of pump B. Boiler C is also provided with a drainage tube 22 for periodically removing material which collects in the bottom thereof.

Pumps A and B are provided with re-entrant receiving bulbs 23 and 24, respectively, for collecting fluid condensed from their respective jets. Pump B is connected to the space undergoing evacuation by means of conduit 25 which communicates with re-entrant bulb 24, while pump A is connected to a suitable backing pump (not shown) by means of conduit 26 communicating with jet 1. The boilers A, B, and C are warmed electrically (by means not shown) and the upper portions of their bulbs may be lagged, if desired, as shown in the device of Figure 5, to conserve heat.

In operation, pump A is connected to the backing pump, while pump B is connected to the space to be evacuated. The pumps are then filled to a depth of one-half to one centimeter with the operating fluid, the backing pump is actuated and heat applied to the boilers.

Assuming the device of Figure 1 to be operating as above outlined, with A serving as the fore pump for B, three ways of working are possible which, for simplicity, may be termed "worst tandem", "normal tandem", and "best tandem".

In the "worst tandem" position, the steel ball 9 is shifted to the left into pocket 10 and condensed fluid from the fore pump A passes through conduit 6, junction 8, pocket 11, and conduit 21 back into the boiler 15 of fine pump B where it is again vaporized and passes back leftwards into pump A. The conditions in worst tandem operation thus correspond to what takes place in the conventional types of pumps, particularly those illustrated in Figures 9, 10, and 11, since practically all of the volatiles are redissolved in the working fluid and flow back into the boiler of pump B. That is, in this manner of operation the volatiles are not kept away from the high vacuum side of the system, but constantly return thereto.

In the normal tandem the steel ball 9 remains in the center depression 12. In this case each pump re-uses its own condensate, that is, condensate from A passes through conduit 6, and pocket 10 back into boiler 14, while condensate from B passes through conduit 7, pocket 11 and conduit 21 back into boiler 15.

In the best tandem the ball 9 is shifted to the right into pocket 11 and condensate then passes from the fine pump B to the boiler 14 of the fore pump A through conduit 7, junction 8, and pocket 10. This fluid is boiled out in the fore pump A, the condensate returning to its own boiler 14 through re-entrant bulb 23, conduit 6, and pocket 10. It will be evident that boiler 15 will constantly lose fluid, but this loss is made up from liquid which returns from the boiler 14 of pump A through conduit 13. Under these conditions, the butyl phthalate filling should be operating under optimum conditions, phthalic anhydride and other "Class B" volatiles collecting in the rings 2, the "Class A" volatiles (gaseous materials) being rejected in the boiler A and withdrawn from the system by the backing pump, the "Class Z" impurities such as the tarry residues and other non-volatile materials drifting from the boiler 15 of pump B through conduit 16 to the evaporator C, while substantially pure phthalate operates under low backing pressure in the fine pump B. It will thus be seen that in this manner of operation the volatiles cannot return to the high vacuum side of the system, but are constantly kept away from that region. Thus the primary object of the invention is accomplished.

The effectiveness of the different methods of operation may be determined by measuring the ultimate vacua obtained. Any satisfactory measurement technique may be employed. For example, the pressure may be measured with an

ionization gauge of the type employing a commercial F. P. 62 tube and the numerical data which follow were obtained by this method. Inasmuch as the use of an ionization gauge for this purpose is well known to those skilled in high vacuum technique, no further details of its operation are included herein.

The readings obtained for the worst-tandem setting of the pump vary with the sequence of testing. In order to secure the minimum difference between the three settings, the pump should be at best-tandem, then at normal-tandem, and finally tested at worst-tandem, thus thoroughly de-gassing the oil in the accepted sense. The three tandem values for butyl phthalate are given in the following table:

Table 1

Oil sequence	At 25° C.		At 0° C.	
	Gauge micro-amps.	Pmm. Hg.	Gauge micro-amps.	Pmm. Hg.
Best-tandem	6	1.5×10^{-4}	.27	6.8×10^{-6}
Normal-tandem	15	3.8×10^{-4}	.45	1.1×10^{-5}
Worst-tandem	280	7.0×10^{-3}	10.0	2.5×10^{-4}

As illustrating the profound improvement obtained by operating a vacuum pump in accordance with my invention, it will be seen from the above table that the best tandem sequence gives a vacuum approximately 40 times lower than that obtainable with the worst tandem sequence.

In examining the quality of vacuum produced by the best tandem setting it was found that very small variations of temperature applied to the tube between the pump and the ionization gauge caused alterations of pressure to be recorded by the gauge. The pressures, and the variations of pressure, were exactly reproducible for butyl phthalate and other homogeneous pump fillings. This fact makes it evident that the optimum vacuum secured with the best tandem setting corresponds with the vapor pressure of the pure fillings at the temperature prevailing on the high vacuum side of the pump.

Oils of the Apiezon type benefit by the best tandem sequence more than the phthalate, because the (C——Y) portion is a graded mixture and the lower boiling members tend towards the fore pump, while the more desirable relatively non-volatile material operates in the fine pump. The results obtained with various types of Apiezon oils at best and worst tandem settings of the experimental pump of Fig. 1 are given in the following table:

Table 2

Kind of oil	Best-tandem			Worst-tandem 25° C.	
	Micro amps.+Pressure mm. Hg.			Micro amps.	Pressure mm. Hg.
	25° C. Ice	25° C.	Ice		
Apiezon A (1934 sample)	3.0 .36	7.5×10^{-8}	9.0×10^{-6}	126.0	3.2×10^{-3}
Apiezon A (1932 sample)	.2 .04	5.0×10^{-6}	1.0×10^{-6}	16.0	4.0×10^{-4}
Apiezon B (1934 sample)	.02 .008	5.0×10^{-7}	2.0×10^{-7}	3.6	9.0×10^{-5}

It will be evident from the above that the simple expedient of operating under such conditions that the volatile constituents of the pumping fluid are constantly kept away from the high vacuum side of the pumping system makes possible the attainment of extremely high vacua with a high degree of facility and without the use of cooling traps involving the use of refrigerants such as carbon dioxide and liquid air and without the use of chemical substances such as charcoal and phosphorous pentoxide. It may be noted in passing that the use of phosphorous pentoxide as suggested in the prior art in connection with certain pumping liquids should never be used with the phthalate esters because the ester molecules are dehydrated by this reagent with liberation of ethylenic bodies and phthalic anhydride.

Having now demonstrated the necessity for providing such fractionating features as will continuously drive the volatile constituents of a filling away from the high vacuum side of a condensation pump, I will now described pumps of simpler design in which this effect is accomplished with varying degrees of completeness.

Figure 2 is a simpler counterpart of Figure 1. In this modification D and E are the fore pump and fine pump respectively, but the compartment C of Figure 1 has been omitted. Where a pump is not to be put to the most exacting or prolonged use it is found that the accumulation of tarry residues is relatively unimportant and a reservoir for these residues may be omitted for the sake of simplicity. In Figure 2 the fine pump is shown with a water jacket **30**, a high vacuum lead **31** and an annulus **32** in which the condensate collects and from which it runs by pipe **33** into the boiler **34** of the fore pump D. This fore pump is fitted with a receiving jet **35** which need not be water cooled, a series of re-entrant fractionating bulbs **36**, an annulus **37** and an intermediate vacuum conduit **38**. Condensate collects in the annulus **37** and passes by tube **39** to the boiler **34**. During operation boiler **40** of the fine pump E tends to become empty and is replenished with fluid from boiler **34** of pump D by way of tube **41**. It is evident that vapor leaving the fine pump jet is derived from liquid that has been boiled out in pump D, the boiling process having driven substantially all the harmful volatiles into the rings **36** or out of the fore pump delivery tube **14**. The pumping fluid is thus able to operate at its best and in its purest condition in the high vacuum fine pump B.

Fig. 3 illustrates a further modification of my invention in which a vertical jet pump F is connected in tandem with, and acts as the backing pump for, an inverted jet pump G. The fore pump F comprises a boiler **50** terminating in a vertical jet tube **51**. The numeral **52** designates a re-entrant bulb for collecting the vapors condensed in the condensing tube **53**. Tube **53** is provided at its upper portion with a series of re-entrant bulbs or rings **54** comprising a miniature fractionating column and also with conduit **55** adapted for connection to a mechanical backing

pump (not shown). Re-entrant collecting bulb 52 is connected to the lower part of boiler 50 by means of drain conduit 56.

Pump G comprises boiler 57 which terminates in jet tube 58. Positioned over, but not closing, the outlet end of tube 58 is the shield or mushroom member 59 providing a means for reversing the direction of the vapors issuing from 58. Tube 58 and member 59 are centrally disposed within a conduit 60 leading from the space undergoing evacuation. Conduit 60 is also provided above the jet with a series of baffles 61 and 62 tending to eliminate diffusion of vapors back into the high vacuum side of the system, the lower portion of conduit 60 forming a condenser for vapors emitted from the jet. Pump G is connected to pump F by means of conduit 64 communicating with the lower end of the conduit 60 and with the re-entrant collecting bulb 52 of pump F.

Pump G is optionally provided with a small evaporator H, comprising a boiler 65 terminating in a re-entrant bulb 66 which in turn is connected by means of conduit 67 to the lower part of conduit 60. Evaporator H is further connected to pump G by means of conduits 68 and 69 communicating, respectively, with boiler 57 and re-entrant bulb 66 and boiler 65. A drainage duct 70 is provided for withdrawing residues which collect in evaporator H from time to time. Boiler 57 of pump G is connected to boiler 50 of pump F by means of return conduit 71. Pumps F and G are supplied with heat, preferably by electrical means (not shown) and the respective boilers may be lagged, if desired, to economize heat.

The operation of the device of Fig. 3 is as follows: Boilers 50 and 57 are filled to a depth of a centimeter or two with the pumping fluid which may be butyl phthalate, or other suitable phthalate ester, or with an oil of the Apiezon type. Conduit 60 of pump G is connected to the vessel or space to be evacuated, conduit 66 is connected to the backing pump, and the latter is actuated. Heat is then applied to the boilers and vapor commences to rise in tube 58 of pump G, passing under member 59 and flowing down along the constricted portion of conduit 60 in the reverse direction, thence through conduit 64 into pump F which operates substantially as described in connection with the corresponding unit of Fig. 1. As pumping progresses the class B volatiles contained in the pumping fluid eventually find their way into the small fractionating column 54 of pump F, the main portion of the fluid being condensed and collecting in re-entrant bulb 52 of pump F from whence it passes by way of conduit 56, boiler 50 and return conduit 71 back to the boiler 57 of pump G. The device is thus operating in accordance with the best tandem sequence of the experimental pump discussed above, the volatiles being withdrawn and kept away from the high vacuum side of the system.

The class Z materials, or relatively non-volatile materials which do not contribute to the pumping effectiveness of the fluid gradually find their way into evaporator H. As heat is applied to this portion of the device, useful pumping constituents are evaporated from the liquid condensed and passed back into boiler 57 by way of conduit 68.

The pumps described in the foregoing description have each consisted of two units working in tandem. Useful and desirable purification of the working fluid can, however, be secured in a single stage. Thus, the fore pump of Fig. 2 performs most of the work of fractionation in tandem assembly. Under appropriate circumstances, the fore pump alone will give efficient service. Thus, when condensible vapors are being handled instead of the permanent gases, the vapors tend to combine with the pump fluid. A simple vertical pump (which may be partly water-jacketed, as shown by the dotted lines of Fig. 2), fitted with a wide fractionating chimney, conveys the vapors from the entrance 58 to the exit 64 (Fig. 2) with greater rapidity and completeness than is achieved by a pump of conventional design.

The device shown in Fig. 4 illustrates another way in which fractionating features, albeit of inferior efficiency, can be incorporated in a single stage pump. The drawing depicts a pump J, of the original Langmuir inverted type shown in Fig. 11, to which has been added a tube 81 conveying vapor to a separate fractionating column 82 the top of which comprises connection 84 communicating with the high pressure pump and with conduit 83 connecting bulb 86 with the backing pump. In operation vapor from the jet 85 condenses and collects in the bulb 86 and passes through the vapor trap 87 to the tube 81 where it passes to the boiler 88 against the stream of hot vapor issuing therefrom. The vapor stream partly purges the condensate of volatiles, and these volatiles, together with other vapors emanating from the boiler itself, pass to the column 82 where the most volatile and harmful constituents are thrust to the top rings of the column 82 and are thus continuously held at the farthest distance from the boiler 88 and thus prevented from interfering with the operation of the pump.

Fig. 5 illustrates a double inverted jet type of single boiler diffusion pump modified to operate in accordance with the present invention. This pump comprises boiler 91 terminating in condensing tube 92 which is directly connected at its upper end to the space to be evacuated. The condensing tube is provided with a centrally disposed jet tube 93 having positioned thereover at a short distance from its open end a mushroom member 94. Tube 93 is also formed at its lower portion so as to provide a second mushroom member 95 cooperating with a similar but somewhat larger centrally disposed tube 96 located underneath. The numeral 97 designates a conduit supplying manifold 98 and perforated tube ring members 99 and 100 with cooling air just above each of the jets.

The high pressure side of the pump comprises the L-shaped conduit 101 provided with a series of re-entrant bulbs 102 constituting a miniature fractionating column. The boiler 91 is lagged with a layer of asbestos wool 103, while the lower part of the tube 92 and the lower part of conduit 101 is lagged with absorbent cotton 104 as shown, the lagging being enclosed within an outer felt jacket 105.

Assuming the pump to be filled to proper depth with the actuating fluid and connected to the space to be evacuated and to the backing pump, the operation is as follows: Heat is applied to the boiler 91 by the electric winding 106, the mechanical backing pump attached to lead 101 is started, and a blast of cooling air is caused to issue from the rings 99 and 100. Presently vapor begins to issue from the mushroom caps 94 and 95 and when a backing pressure of less than .1 mm. is reached the pumping action comes into full operation. Contrary to usual practice in which a water jacket is employed, care is taken to cool the condensing walls 92 only partially. Similarly, in

ordinary pumps of this inverted type, precautions are taken to prevent condensate from touching the hot vapor pipe at its base 107. In the present pump, the survival of uncondensed vapor, or the regeneration of vapor from contact of the condensate with the hot parts, is encouraged; and the rate of heating and the degree of cooling as well as the quantity of lagging is adjusted to secure a gentle stream of vapor towards the fractionating rings 102 in the lead 101. This stream of vapor is adjusted to be of such a density that it adds inappreciably to the resistance of gases passing through the pump, yet is amply sufficient to convey any unwanted volatiles to the fractionating rings. The size and number of these rings is so adjusted that none of the true pumping fluid escapes from the pump. More volatile materials, such as are frequently entrained or extracted from gases entering the high vacuum lead 92 are continually thrust away from the boiler and tend to accumulate in the upper rings of the fractionating lead. It is sometimes convenient to attach a reservoir to the overflow tube 108 to accumulate the excess of condensate.

While a three compartment pump such as shown in Figure 2 segregates the A, B, and Z impurities fairly well, it cannot arrange in order the C——Y constituents of a mixed pump filling. In view of the above discussion of the experimental three compartment pump of Fig. 1, it is evident that a mixed pump filling could be put to great advantage if the pumping fluid could be utilized in such manner that the lower boiling, high vapor pressure fractions did most of the pumping at the high pressure end of the system, while the higher boiling, low vapor pressure fractions would be utilized at the low pressure end and thus reduce the residual gas pressure to an extremely low value. I have found that this desirable result can be attained by the devices illustrated by way of example in Figures 6 and 7, which may be referred to as multi-stage cascade pumps.

In the device illustrated in Figure 6, the numeral 111 designates a metal box, rectangular in plan, wedge-shaped in elevation. Around the edge of the open top is brazed a stout steel flange 112, the upper surface of which is lapped to receive a lid, conveniently a sheet of plate glass 113. The plate 113 forms the lower element of a transparent water-cooled cell 114, the upper element being another sheet of plate glass 115. The cell is supplied with cooling water by means of inlet conduit 116 and emptied by means of outlet conduit 117.

The inside of the box 1 is provided with a number of bent partitions 118, 119, 120, 121, 122, 123, 124 and 125, optionally soldered or welded to the sides thereof, the lower ends of these partitions terminating about one millimetre from the floor 126. The horizontal portion of the partition 118 forms with the corresponding horizontal partition 119, etc., a series of jets, each a little narrower than the one preceding it from left to right.

The low pressure end of the device communicates with the space to be evacuated by means of conduit 127, while the high pressure end of the device is provided with a conduit 128 communicating with the jet chamber, the vertical portion of this conduit being optionally provided with a series of re-entrant bulbs or rings 129 and with a conduit 130 adapted for connecting to a mechanical backing pump (not shown).

The pump 111 is mounted on a conventional type of electrical heater 131, heat being supplied by means of a current passing through resistance coil 132 conveniently tapped in three places so that extra heat can be applied to the incoming oil. The whole assembly rests on a table 133 which can be tilted by means of a leveling screw 134.

Fig. 7 is a perspective view of a device substantially identical with that of Fig. 6, except that an additional cooling jacket 135 is provided at the high vacuum intake.

Although I have found it convenient to illustrate this type of pump as one provided with seven graduated compartments and jets the number has no particular significance, as it is obvious that some types of pumping fluids will require more and some a less number of compartments, depending upon how widely separated are the boiling points of the constituents of the selected fluid.

The operation of the multiple-stage cascade pump is as follows: Assuming by way of example, that the pump is filled with a mixture of equal parts of normal amyl phthalate (approximate boiling point 164° C. at 1 mm.) and β-phenyl ethyl phthalate (boiling point 237° C. at 1 mm.) representing an extreme case, the heat input is adjusted to maintain a uniform temperature of about 170° C. along the bottom of the pump during operation. Conduit 127 is connected to the space to be evacuated and conduit 130 is connected to the mechanical backing pump and the latter is actuated. Under vacuum the amyl ester will begin to evaporate from the mixture and issue from the jets, condensing on the sloping glass ceiling 113 down which it runs into the high pressure end of the pump and thence under the lower end of partition 125 into compartment K. The contents of K are now richer in amyl phthalate and vigorous distillation ensues, giving a strong pumping action at the jet *k*. Distillation is going on though to a lesser extent in compartment L, so that the level of liquid falls, its place being taken by mixture from compartment K which has lost some of its amyl phthalate. In the same way, compartment M draws liquid from compartment L, N from M, and so on, until P and Q receive liquid which is substantially free from amyl phthalate, and is seven times purified from air and other volatile constituents.

The mixed vapors from or around the region of jet *n* should have reduced the pressure to below 10^{-5} mm. so that the β-phenyl ethyl ester, which fails to boil without decomposition in the three compartment pump of Fig. 2, can evaporate in the latter compartments of the pump illustrated in Fig. 6 at a safe, low temperature and provide a gentle stream of low pressure vapor through a short wide jet system to secure the final improvement of the vacuum.

One of the outstanding features of my invention as embodied in the cascade pump just described is that unpurified pump fillings can be used. During the preparation of complex esters such as butyl benzyl phthalate, for example, a mixture containing this substance with both the di-butyl and di-benzyl esters is produced from which it is difficult to separate the individual components. This mixture can be used without purification in the cascade pump of Figures 6 and 7, since the various constituents of the fluid soon take up their appropriate working positions in the system. The same general principle holds true for many mixtures of the phthalate esters,

the Apiezon oils and many other multiple-component operating fluids.

It will, of course, be evident that many modifications may be made in the method of, and the apparatus for, carrying out the above described technique within the scope of my invention. While I have illustrated the principles underlying the invention by reference to pumps comprising a definite number of units, their number may be multiplied as desired. It will also be evident that the device herein described may be constructed of any suitable material, such as glass, metal, and the like in accordance with the demands of any particular uses to which they are applied. Likewise the matter of size is not critical and the pumps may be designed to meet the requirements of any given commercial operation.

The pumping technique and apparatus herein described may be applied to a wide variety of uses. It will be found particularly valuable in obtaining the high vacua required in various vacuum or molecular distillation operations. The invention finds an especially important application in the manufacture of X-ray and other types of vacuum tubes and in the electric lamp industry in general, since it provides a simple and reliable method for exhausting such receptacles to almost any desired degree of vacuum. The invention is likewise of a special importance to research workers in the high vacuum field.

What I claim is:

1. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, means for producing a stream of vapor, a condensing element, at least one jet through which the stream of vapor flows to the condensing element, means for returning condensate from the condensing element to the vapor producing means, a conduit communicating with the jet and ultimately with the space being exhausted, a conduit communicating with the condensing element and a source of suction, and means located on the low vacuum side of the pump for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump.

2. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, means for producing a stream of vapor, a condensing element, a jet through which the stream of vapor flows to the condensing element, means for returning condensate from the condensing element to the vapor producing means, a conduit communicating with the jet and ultimately with the space being exhausted, a conduit communicating with the condensing element and a source of suction, and means on the low vacuum side of the pump for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump, said last named means comprising a fractionating element forming a part of the conduit connecting the condensing element with said source of suction.

3. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, a boiler for producing a stream of vapor, a condensing tube, a jet through which the stream of vapor flows to the condensing tube, a conduit for returning condensate from the condensing tube to the boiler, a conduit communicating with the jet and ultimately with the space being exhausted, a conduit communicating with the condensing tube and a source of suction, and a miniature fractionating column forming a part of the last named conduit for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump.

4. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, a boiler for producing a stream of vapor, a condensing tube, a jet through which the stream of vapor flows to the condensing tube, a conduit for returning condensate from the condensing tube to the boiler, a conduit communicating with the jet and ultimately with the space being exhausted, a conduit communicating with the condensing tube and a source of suction, and a miniature fractionating column forming a part of the last named conduit for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump, and means connected to the boiler for separating and segregating phlegmatic residues from the pumping fluid whereby said residues may be removed from the boiler and useful components returned in vapor form to the vapor stream.

5. A vacuum pumping unit of the diffusion type adapted to employ a mixed organic liquid as a working fluid comprising a series of jet pumps connected in tandem, separate means for producing a stream of vapor through each jet, a condensing tube in proximity to each jet for condensing the vapors passing therethrough, means communicating with the condensing tubes for returning condensate to the vapor producing means, a conduit connecting the first pump of the series to the space to be evacuated, a conduit connecting the last pump of the series with a source of suction, said last named conduit being provided with a fractionating element for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump.

6. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, a series of jet pumps connected in tandem, each pump acting as a fore pump for the pump preceding it and comprising a boiler for producing a stream of vapor, a condensing tube, a jet through which said stream of vapor flows to said condensing tube, a receptacle for collecting condensate from the condensing tube, a conduit for returning condensate from the condensing tube to the boiler, a conduit connecting each condensing tube with the collecting receptacle of the succeeding pump; a conduit for connecting the last pump of the series with a source of suction; a conduit connecting the first pump with the space to be evacuated, conduits communicating with and connecting the boilers of the respective pumps; a fractionating element communicating with the collecting receptacle and boiler of the first pump for separating and segregating phlegmatic residues from the working fluid whereby said residues are withdrawn from the pump, and a fractionating element located on the conduit connecting the last pump with a source of suction for separating and segregating volatiles from the

pumping fluid whereby the more volatile components are withdrawn from the pump.

7. A vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a working fluid, comprising in combination, a series of jet pumps connected in tandem, each pump acting as a fore pump for the pump preceding it and comprising a boiler for producing a stream of vapor, a condensing tube, a jet through which said stream of vapor flows to said condensing tube, a receptacle for collecting condensate from the condensing tube, a conduit for returning condensate to the boiler, a conduit connecting each condensing tube with the collecting receptacle of the succeeding pump; a conduit for connecting the last pump of the series with a source of suction; a conduit connecting the first pump with the space to be evacuated, conduits communicating with and connecting the boilers of the respective pumps, a fractionating element communicating with the collecting receptacle and boiler of the first pump for separating and segregating phlegmatic residues from the working fluid whereby such residues are prevented from returning to the pump; a fractionating element located on the conduit connecting the last pump with a source of suction for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing return of said volatiles to the high vacuum side of the pump and valved tubular means connected to the collecting receptacles and boilers of said pumps and to the separating device for the purpose of controlling vapor and condensed materials in the pumps.

8. A multi-stage fractionating vacuum pump of the diffusion type adapted to employ a mixed organic liquid and in operation to arrange the components thereof so that the low vapor pressure components exert their pumping action toward the high vacuum side and the high vapor pressure components toward the low vacuum side of the pump, comprising in combination, a vessel forming a horizontal jet chamber, a series of jets of progressively decreasing cross-sectional area located within said chamber, a common condenser for all of the jets, a reservoir supplying fluid to all of the jets, means for conveying condensate from the condenser to said reservoir, a conduit for connecting the space to be evacuated with the larger jet and a conduit located at the smallest jet for connecting to a source of suction.

9. A multi-stage fractionating vacuum pump of the diffusion type adapted to employ a mixed organic liquid as a pumping fluid and in operation to arrange the components thereof so that the low vapor pressure components exert their pumping action toward the high vacuum side and the high vapor components toward the low vacuum side of the pump, comprising in combination, a vessel forming a horizontal jet chamber, a plurality of partitions within said chamber arranged to form a series of separate vaporizing compartments and jets of progressively decreasing cross-sectional area, a common condenser positioned in said chamber in proximity to said jets and functioning to condense vapors emitted therefrom, a reservoir within the chamber supplying fluid to all of the vaporizing compartments, means for conveying condensate from the condenser to said reservoir, and inlet and outlet ports at the largest and smallest jets respectively.

10. A multi-stage diffusion pump adapted to employ a mixed organic pumping fluid and in operation to arrange the components thereof so that the low vapor pressure components exert their pumping action toward the high vacuum side and the high vapor pressure components toward the low vacuum side of the pump, comprising in combination, a hollow vessel having integral bottom, side and end walls forming a jet chamber, the top wall comprising an inclined common condenser for a plurality of jets, a series of right-angle partitions sufficiently spaced from the bottom of said chamber to permit the passage of a liquid thereunder, said partitions being arranged with respect to the top wall and in substantial parallel relation one to another to form a series of vaporizing compartments and jets of progressively decreasing cross sectional area from the high vacuum to the low vacuum side of the pump, a common sump for pumping fluid communicating with each of said compartments and located opposite the condenser, a conduit for conveying condensate form the condenser to the sump, and inlet and outlet ports at the largest and smallest jets respectively.

11. A horizontal, multi-stage diffusion pump adapted to automatically segregate the components of a mixed organic pumping fluid, comprising a hollow vessel having integral bottom, side and end walls forming a jet chamber, the top wall comprising two substantially parallel inclined plates spaced apart to provide a passage for a cooling fluid there-between and acting as a condenser for a plurality of jets, a series of right-angle partitions supported within and spaced sufficiently from the bottom of the chamber to permit the passage of a liquid under their lowermost edges, said partitions being arranged in substantially parallel relations one to another to form a series of vaporizing compartments and jets of progressively decreasing cross-sectional area from the high vacuum to the low vacuum side of the pump, a common sump for pumping fluid communicating with each of said compartments and located opposite the condenser, a conduit for conveying condensate from the condenser to the sump, and inlet and outlet ports at the largest and smallest jets respectively.

12. In the process of evacuating a chamber by means of a diffusion or condensation pump employing an organic pumping fluid containing or tending to form volatiles the steps which comprise, constantly fractionating the vaporized pumping fluid and separating volatile components therefrom which would otherwise return to the boiler of the pump and returning the fluid, stripped of the more volatile fractions, to the pump for re-use.

13. In the process of evacuating a chamber by means of a diffusion or condensation pump employing an organic pumping fluid containing or tending to form volatiles the steps which comprise constantly fractionating the pumping fluid at the low vacuum zone of the pump and separating volatile components therefrom which would otherwise return to the boiler of the pump and returning the pumping fluid stripped of the more volatile constituents to the pump boiler for re-use.

14. The method of producing a high vacuum which comprises heating and vaporizing an organic pumping fluid containing or tending to form volatiles, projecting a stream of the vapors through a jet, admitting gas from the space to be exhausted into the jet of vapors, condensing and fractionating the jet vapors at a remote point and separating volatile components therefrom which would otherwise return to the boiler

of the pump and returning the condensed pumping fluid stripped of its more volatile fractions to the heating zone for re-use.

15. In the process of evacuating a chamber by means of a series of diffusion or condensation pumps employing a mixed organic pumping fluid and having individual boilers the steps which comprise constantly fractionating the pumping fluid and circulating the segregated fractions to individual pumps, the lowest vapor pressure fractions being circulated to the boiler of the lowest pressure condensation pump and the most volatile to the highest pressure condensation pump.

16. A condensation pump comprising in combination, means for producing a stream of vapor of an organic evacuating liquid, a condensing element, a jet forming means through which the vapor stream flows into the condenser, a conduit communicating with the jet of vapors and ultimately with the chamber to be evacuated, means for returning condensed evacuating liquid to the vapor generating means, and means for separating and segregating, from the evacuating liquid, volatile constituents which would otherwise be repeatedly returned to the vapor producing means.

17. A vacuum pump of the diffusion type adapted to employ an organic liquid as a working fluid, comprising in combination, means for producing a stream of vapor, a condensing element, at least one jet through which the stream of vapor flows to the condensing element, means for returning condensate from the condensing element to the vapor producing means, a conduit communicating with the jet and ultimately with the space being exhausted, a conduit communicating with the condensing element and a source of suction, and means for separating and segregating the more volatile components, condensed in the condensing zone, from the pumping fluid and preventing said volatiles from diffusing into the high vacuum side of the pump.

18. In the process of evacuating a chamber by means of a diffusion or condensation pump employing an organic pumping fluid containing or tending to form volatiles the steps which comprise, constantly separating volatile components therefrom which would otherwise return to the boiler of the pump and returning the fluid stripped of the more volatile fractions, to the pump for re-use.

KENNETH C. D. HICKMAN.