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K. C. D. HICKMAN
METHOD OF EVACUATING
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1,857,508

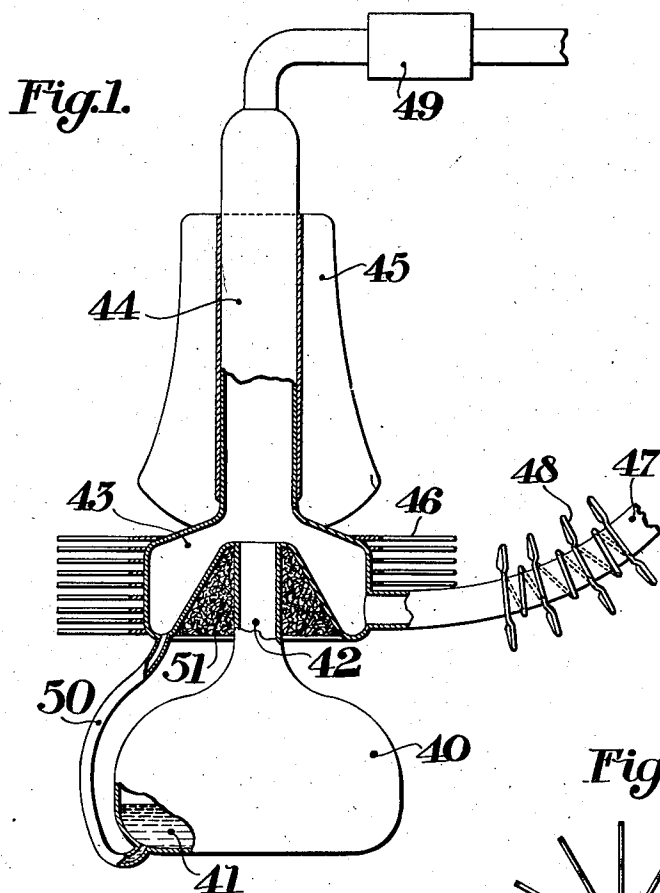


Fig. 2.

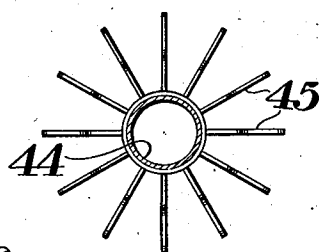


Fig. 3.

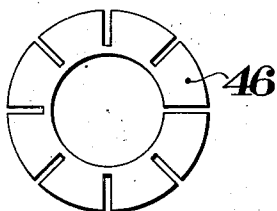
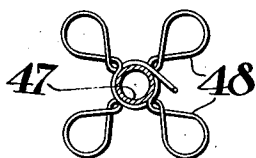


Fig. 4.



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METHOD OF EVACUATING

Application filed April 4, 1931. Serial No. 527,714.

My present invention relates to a method of evacuating an enclosed space by means of organic liquids of low volatility and decomposition rate, such as the esters of phthalic acid. The organic liquid is evaporated by heat and the vapors are passed through a jet thus ejecting them from the evaporating flask at high velocity. These vapors envelop or enclose the gases with which they contact and carry them along away from the point of contact, thus continually removing the gases from a certain area. By attaching the closed vessel to be evacuated to this area with which the current of organic vapor contacts, removal of the gases contained therein and consequent diminution of pressure is accomplished. The vapors are in practice condensed and returned to the evaporating flask to diminish back pressure which might seriously impair the efficiency of the process.

The present application is in part a continuation of my application Serial No. 341,105 filed February 19, 1929.

In the past in the various types of vacuum pumps which have relied on a current of vapor as the evacuating agent, mercury has been employed for the reason that it is an elementary substance and consequently will not break down into gaseous or objectionable compounds. Also its position in the electromotive series is indicative of its resistance to acidic materials. However mercury has a high surface tension and when it condenses it gathers together in globules or droplets instead of distributing itself over the condensing surface. Consequently when the atoms of the mercury vapor hit the condensing surface some of the mercury which hits the surface not wetted by a liquid will deflect or bounce from the surface before condensation can take place. This has the effect that the total area of the condensing surface is not effective in inducing condensation.

Mercury has another disadvantage in the vapor type vacuum pumps, in that the gases carried forward by the vapor stream may leak backwards between globules of the vapor rendering the pressure on the high vacuum side of the pump a definite function of that

on the lower side, instead of, letting it be within limits, independent thereof. In the absence of an effective backing pump to cooperate with the vapor-type pump when it contains mercury, the effectiveness of the vapor-type evacuation pump is considerably reduced.

A further disadvantage of mercury is that its vapor pressure at ordinary temperatures may be as high as a micron (10^{-3} m. m.) of mercury so that it is necessary to interpose a low temperature trap to prevent diffusion of vapor from the pump to the vessel being evacuated, when the highest vacuums are desired. An example of a low-temperature trap that may be used is a U-tube cooled by liquid air.

I have found that organic liquids having certain characteristics are particularly suitable for employment in the vapor-type vacuum pump. The difficulty volatile organic liquids which I have found especially suitable are the esters of the phthalic acids such as n-butyl phthalate, iso amyl phthalate, n-butylterephthalate, n-amylphthalate, butyl benzyl phthalate or like esters of phthalic acid. The organic liquids that have been found most suitable in this connection are the phthalic, isophthalic or terephthalic acid esters of the methyl, ethyl, propyl, butyl, amyl or benzyl alcohols either normal or isomeric. The characteristics these suitable liquids possess are:

(1) A vapor pressure at about 0° C. which is less than that encountered in industrial high vacuum work.

(2) The property of condensing easily as a film on a cooled surface.

(3) They may be boiled for long periods without chemical decomposition.

(4) They are chemically inert in so far as the material of the pump and the gases, which are usually met with in vacuum work, are concerned.

I have found that the many disadvantages of mercury are overcome to a large extent by using certain high boiling organic liquids in vapor-type vacuum pumps in place of mercury. Although low vapor pressure is the most important criterion of the suitability

of a liquid, obviously high boiling liquids which are decomposed by boiling are not adapted to be employed in this connection. Certain aliphatic liquids such as the higher paraffins, and the long chain acids and esters, both saturated and unsaturated, although they do eventually produce high vacua when used in vapor type vacuum pumps, do so comparatively slowly, probably because of the cracking of these compounds by heat, into more volatile constituents. If the cracking of these compounds is the cause of diminution of speed of evacuation, the reason for this seemingly lowered efficiency is apparently due to the fact that the pump must perform the double duty of removing the gas from the vessel being evacuated and also of removing the more volatile waste products of the cracked vapor.

On the other hand, the esters of the phthalic acids which I employ have a sufficiently low vapor pressure at room temperature that they may even be used without a "liquid air" trap previously referred to. These liquids have a low surface tension and will wet the condensing surface so that this surface is effective over its whole area in inducing condensation and a shorter pump may be used as the vapor condenses within a well defined zone. This behavior is in contrast with that of mercury. When mercury is employed its vapor wanders out of the pump and is found deposited in various corners and bends. In the vacuum pumps employing mercury it has been necessary to provide a condensing system of relatively great length to lessen the quantity of mercury vapor that might enter the vessel which is being evacuated.

However, the method which I have invented may be carried out in any compatible type of vapor vacuum pump such as, for example, the pump disclosed in U. S. Patents Nos. 1,320,874 and 1,393,550 of Langmuir or in the pumps disclosed in my co-pending application No. 341,105. As a specific example of the carrying out of this process, it is described with reference to the vacuum pump shown in Figure I which is claimed in my co-pending application No. 341,105.

Referring to the drawings, in which like reference characters refer to like parts, Figure I is one form of evacuation pump in which my process may be carried out. Figures II, III, and IV are cross-sectional views of the pump depicted in Figure I, taken on lines 2-2, 3-3 and 4-4 thereof, respectively.

Referring to the drawings which illustrate apparatus suitable for carrying out my process, Figure I shows a pump which comprises a vessel 40 containing the organic liquid such as an ester of phthalic acid, 41, which is to be boiled by any suitable source of heat applied thereto. The vessel 40 is constricted into a jet 42 connected with a reentrant bulb 43 which is distorted into the form of an ex-

aggerated alembic which is connected to a condensing tube 44. Bulb 43 is also connected to the vessel to be evacuated through tube 47.

The condensing tube 44 may be cooled by means of a radiator 45 in the form of a metal collar provided with metallic radiating fins engaging the tube 44 as shown in Figure II.

A heat radiator, 46, may also be provided around bulb, 43, preferably formed of separate fins such as that shown in Figure III. The tube, 47, may also be provided with a cooling device. The cooling device may consist of spaced turns of wire, 48, as shown in detail in Figure IV.

The space between jet, 42, and the bulb 43 is packed with a heat insulating material, 51, to prevent transfer of heat from the jet to the bulb or chamber. Also the condensing tube 44 is connected to the atmosphere preferably with a backing pump, 49, interposed.

In the operation of my process, the organic liquid, 41, which may be butyl phthalate is boiled and a stream of its vapor rapidly passes out through the jet 42 into condensing tube 44. The current of vapor which passes from jet 42 into tube 44 entraps the air or gas which is present in bulb, 43. By this means the pressure in bulb 43 is continuously lowered, which simultaneously lowers the pressure of the entire enclosed system represented by tube 47.

When the vapor reaches the tube, 44, it condenses as a film, and drops back into bulb, 43, where it cools and collects and then passes through return tube, 50, into the container, 40. Here it is heated and again goes through the cycle of the operation.

With the process of my application a 24 liter flask has been exhausted from a pressure of 2 m.m. of mercury to that of .000003 m.m. of mercury in 15 minutes, thus illustrating the speed and efficiency of my process.

It is understood that instead of the individual organic liquids, mixtures of various liquids which are compatible with the details of the invention may be used instead of the individual compounds.

What I claim as my invention is:

1. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises removing a portion of the entrained gases in the vessel to be evacuated by means of a current of phthalic acid ester vapors.

2. A method of evacuating a closed receptacle to obtain a high vacuum therein which comprises removing a portion of the entrained gases in the closed receptacle by means of the vapor of an ester of phthalic acid selected from the group consisting of normal-butyl phthalate, iso-amyl phthalate, normal-butyl terephthalate, normal-amyl phthalate, and butyl benzyl phthalate.

3. The method of evacuating a closed re-

ceptacle to obtain a high vacuum therein which comprises removing a part of the entrained gases in the closed receptacle by means of the vapor of normal-butyl phthalate.

12. A means for evacuating closed vessels which comprises a diffusion and condensation vacuum pump containing n-butyl phthalate as an evacuating medium.

Signed at Rochester, New York, this 25th day of March, 1931.

KENNETH C. D. HICKMAN.

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| 6 | 4. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises removing a part of the entrained gases in the closed receptacle by means of the vapor of iso-amyl phthalate. | 75 |
| 10 | 5. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises removing a part of the entrained gases in the closed receptacle by means of the vapor of butyl benzyl phthalate. | 80 |
| 15 | 6. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises vaporizing an ester of phthalic acid selected from the group consisting of normal-butyl phthalate, iso-amyl phthalate, normal butyl terephthalate, normal-amyl phthalate and butyl benzyl phthalate, projecting a stream of said vapors through a restricted jet connected with the receptacle to be evacuated, condensing the vapor at a remote point, re-evaporating the condensed vapor and introducing it into the moving stream of vapor. | 85 |
| 20 | 7. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises vaporizing normal-butyl phthalate, projecting a stream of said vapors through a restricted jet connected with the receptacle to be evacuated, condensing the vapor at a remote point, re-evaporating the condensed vapor and introducing it into the moving stream of vapor. | 90 |
| 25 | 8. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises vaporizing iso-amyl phthalate, projecting a stream of said vapors through a restricted jet connected with the receptacle to be evacuated, condensing the vapor at a remote point, re-evaporating the condensed vapor and introducing it into the moving stream of vapor. | 95 |
| 30 | 9. The method of evacuating a closed receptacle to obtain a high vacuum therein which comprises vaporizing butyl benzyl phthalate, projecting a stream of said vapors through a restricted jet connected with the receptacle to be evacuated, condensing the vapor at a remote point, re-evaporating the condensed vapor and introducing it into the moving stream of vapor. | 100 |
| 35 | 10. A means for evacuating closed vessels which comprises a diffusion and condensation vacuum pump containing an ester of a phthalic acid as an evacuating medium. | 105 |
| 40 | 11. A means for evacuating closed vessels which comprises a diffusion and condensation vacuum pump containing an alkyl phthalate as an evacuating medium. | 110 |
| 45 | | 115 |
| 50 | | 120 |
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| 60 | | 130 |