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HIGH VACUA

James G. Baxter, Rochester, N. Y., assignor, by mesne assignments, to Distillation Products, Inc., Rochester, N. Y., a corporation of Delaware

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This invention relates to improvements in method and apparatus for the production of high vacua and more particularly to improved condensation pump fluids.

It is an object of this invention to provide new and improved fluids for use as actuating media in a condensation or diffusion pump. Another object is to provide improved methods of producing high vacua. A still further object is to provide improved pumping means. Other objects will become apparent from the following description.

In the past mercury was exclusively employed as the actuating fluid in a condensation or diffusion pump. Recently esters of phthalic acid were found to have superior pumping properties and they have practically displaced the use of mercury for this purpose. Pump fluids must possess two properties in particular. Their vapor pressure at the operating temperature must be low and the amount of decomposition which they suffer during use must be slight. Until the advent of my invention the esters of phthalic acid were the only pure compounds available with these characteristics.

I have discovered that di-esters of aliphatic dibasic acids containing more than four carbon atoms have superior properties as condensation pump fluids. It has also been found that the di-alkyl esters of any aliphatic dibasic acid have exceptional pumping properties. Di-esters which contain at least 12 but not more than 42 carbon atoms are preferred.

Examples of suitable aliphatic dibasic acids are glutaric, adipic, pimelic, suberic, azelaic and sebacic acids. The esters of sebacic acid are of particular value. These and other aliphatic dibasic acids may be esterified with any alcohol, but preferably with an aliphatic alcohol. Examples of such alcohols are ethyl, butyl, amyl, hexyl, heptyl, n-octyl, 2-ethyl hexyl and nonyl. Mixtures of 2 or more alcohols may be used to yield a mixed ester, if desired. Sebacates having the formula,

ROOC $CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2COOR'$

in which R and R' represent hydrocarbon and preferably alkyl radicles of 4 to 9 carbon atoms, are most useful for the production of intermediate and high vacua. Specific examples of esters are di-lauryl oxalate, di-capryl succinate and adipate, di-amyl, -butyl, -hexyl, -2 ethyl hexyl sebacate, di-(β phenyl ethyl) and di-(γ phenyl propyl) adipate.

The particular combination selected will largely depend upon the degree of vacua to be produced. Esters having a lower number of carbon atoms, as a general rule, have a higher vapor pressure. They are therefore most useful for lower vacuum work, such as in the booster pumps, described in the co-pending Hickman application #130,359 filed March 11, 1937. The higher molecular weight esters have a lower vapor pressure and are most useful for the production of the highest vacua. Esters suitable for all such purposes will usually contain 12 to 42 carbon atoms in the molecule. Those having a boiling point of between about 150° and 180° C. at .1 mm. have been found to be best for the latter purpose.

The esters can be prepared by known esterification methods. One such method which gives high yields is to mix one molecular portion of the particular acid selected with an equal weight of toluene and 2–3 molecular proportions of the appropriate alcohol. This mixture is stirred until a clear solution results and 1% of p-toluene sulfonic acid catalyst, based on the weight of acid is added. The mixture is then heated to distillation temperature in a flask provided with a condenser. Water formed during the reaction distills with the toluene and the mixture is condensed, stratified and the toluene returned to the reaction flask. Practically complete esterification takes place in about 4 hours, while after 8 hours heating, less than .1% free acid remains. Toluene and unreacted alcohol are removed by distillation at water pump pressure and the residual acid, by distillation at about .1 mm. pressure. The esters prepared in this way are odorless and water-white in color. Any other known method of preparing the esters can, of course, be used.

A particular advantage of esters of aliphatic dibasic acids is the ease with which the ester can be produced. It will be noted that the esterification reaction requires much less time than usual and goes practically to completion in the short time of 4 hours. For this reason little free acid remains and purification of the reaction mixture to the high degree necessary for pumping fluids is considerably simplified.

The esters described have to a remarkable degree, the desirable properties of a superior pumping fluid, namely a low vapor pressure at operating temperature and high resistances to thermal decomposition and oxidation during use. In addition they have a higher boiling point than the corresponding phthalates and a simpler trapping device can therefore be used. For instance 2-ethyl hexyl sebacate will produce a vacuum

of 1.9×10^{-8} at 25° C. in a 3 compartment condensation pump as compared with 5×10^{-7} for 2-ethyl hexyl phthalate ("octoil"). It boils at 175° C. at .1 mm., some 20° C. higher than the boiling point of "octoil," without any apparent increase in the rate of decomposition. It is also markedly more stable to air at high temperature, and therefore offers a greater degree of protection from the accidental intrusion of air during operation of the pump. The butyl and amyl sebacates are likewise superior to the corresponding phthalates and may be expected to replace them where the highest vacua are not needed. In general, however, the great thermal stability which the esters of aliphatic di-basic acids possess, constitutes their superiority over the corresponding phthalates now in use as pumping fluids in condensation pumps.

The esters described can be employed in any type of condensation pump, but preferably are used in condensation pumps designed for organic working fluids. The construction or design of such pumps is well known in the art and does not form a part of this invention.

What I claim is:

1. Means for evacuating a closed system which comprises a condensation pump containing as a working fluid a di-alkyl ester of an aliphatic dibasic acid, the ester being one which contains at least 12 but not more than 42 carbon atoms.

2. Means for evacuating a closed system which comprises a condensation pump containing as a working fluid a di-ester of an aliphatic dibasic acid, the ester being one which has a boiling point of between about 150° and 180° C. at .1 mm.

3. Means for evacuating a closed system which comprises a condensation pump containing a di-ester of sebacic acid as a working fluid, the ester being one which contains at least 12 but not more than 42 carbon atoms.

4. Means for evacuating a closed receptacle which comprises a condensation pump containing a compound having the following formula, as a working fluid:

$$ROOC\ CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2COOR'$$

in which R and R' are hydrocarbon radicles containing 4 to 9 carbon atoms.

5. Means for evacuating a closed receptacle which comprises a condensation pump containing as a working fluid, di-2-ethyl hexyl sebacate.

6. Means for evacuating a closed receptacle which comprises a condensation pump containing as a working fluid a di-ester of an aliphatic di-basic acid, the di-basic acid being one which contains at least four carbon atoms and the di-ester being one which contains at least twelve but not more than forty-two carbon atoms.

JAMES G. BAXTER.