

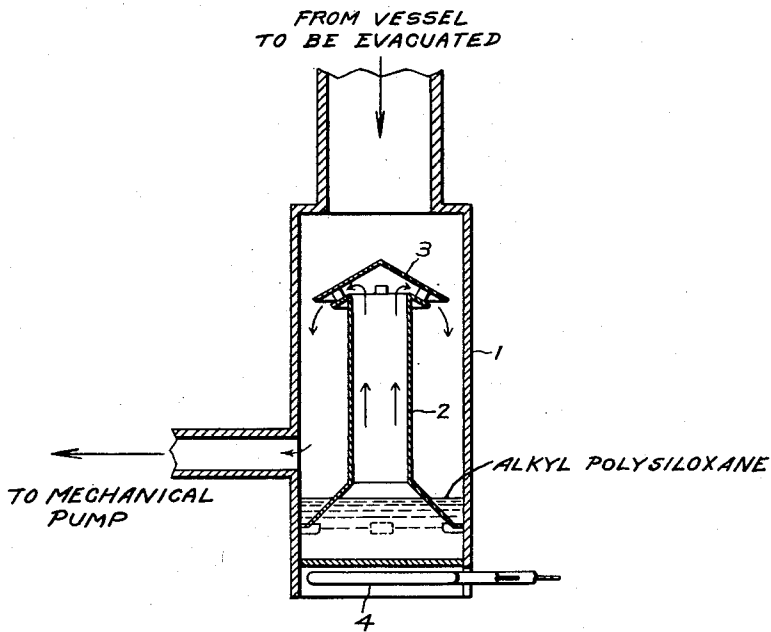
May 10, 1949.

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2,469,889

VACUUM PUMP

Filed June 28, 1945



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UNITED STATES PATENT OFFICE

2,469,889

VACUUM PUMP

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Application June 28, 1945, Serial No. 602,158

9 Claims. (Cl. 230-101)

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The present invention relates to the production of high vacua by means of condensation pumps, and more particularly to improved condensation pump fluids.

Condensation or diffusion pumps have been used for many years to evacuate closed systems or receptacles by removing a portion of the entrained gases in the closed systems. For this purpose, various liquids have been suggested or used as the pumping or actuating fluid in such pumps. Mercury, which was one of the first so employed, has as its principal advantage good heat stability. However, as it also has certain disadvantages, including a substantial vapor pressure at ordinary temperatures as measured by modern requirements for such fluids, it has been largely supplanted by various organic esters and highly refined hydrocarbons which in general possess better pumping properties and lower vapor pressures than mercury at ordinary temperatures. Unfortunately these organic compounds tend to decompose or oxidize during use with the formation of both lower boiling or volatile products and higher boiling tarry residues. To overcome the difficulties resulting from the formation of the volatile decomposition products, relatively complex "fractionating" condensation pumps have been devised for the purpose of separating or removing these products so that they will not interfere with the operation of the undecomposed fluid. However, no means has been devised for successfully removing the tar-like decomposition products which contaminate the working fluid and collect on the condensing surfaces of the pump. These residues reduce the overall efficiency of the pump necessitating frequent cleaning of the pump and replacement of the working fluid.

The present invention is based on the discovery that certain liquid lower alkyl polysiloxanes, particularly the methyl polysiloxanes or mixtures thereof, containing at least nine silicon atoms per molecule have superior properties as condensation pump fluids. As the source of the propellant vapor in condensation pumps, these polysiloxanes have been found to combine all of the operating advantages of the low vapor pressure organic fluids plus a heat stability which under normal pumping conditions is comparable to that of mercury. They are superior to the organic materials in that they are completely inert towards oxidation up to temperatures of 150° C. and highly resistant to oxidation up to 200° C. They are likewise resistant to cracking up to 200° C. and may be used successfully without cold traps. The

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operational life of the polysiloxane fluids has been found to be many times that of the hydrocarbon or ester-type fluids, thus eliminating costly shutdowns for cleaning the pump and changing the operating fluid.

The particular liquid polysiloxane or polysiloxane mixture selected will depend upon the degree of vacuum desired and in some cases on the type of equipment employed. In general the methyl polysiloxanes are preferred because of their superior oxidation resistance as compared with the ethyl or propyl polysiloxanes and the invention will be specifically described with reference to the methyl polysiloxanes. As a phenyl group attached to silicon is also relatively stable, certain liquid alkyl phenyl polysiloxanes containing both phenyl and methyl or other lower alkyl radicals can also be employed and the term "alkyl polysiloxane" as used herein and in the appended claims is intended to include such a hydrocarbon-substituted polysiloxane containing a major portion of silicon-bonded lower alkyl radicals and a minor portion of silicon-bonded phenyl radicals in its molecular structure.

For a given series of liquid polysiloxanes, those having the greater number of silicon atoms have the lower vapor pressures at room temperature. However, as they also have higher boiling points, the upper limit in the number of silicon atoms will depend on the boiling points of the polysiloxane or polysiloxane fraction. In general, the straight or branched-chain polysiloxanes are preferred over the cyclic polysiloxanes as pumping fluids since the chain compounds have somewhat lower vapor pressures at room temperature than the cyclic polysiloxanes of the same boiling points. Because mixtures of the chain compounds or mixtures thereof with cyclic compounds are usually obtained in the preparation of the liquid polysiloxanes useful in the practice of the present invention, such mixtures are usually employed commercially to avoid the expense of isolating the individual compounds.

The classes of liquid polysiloxanes of which those employed in the present invention form a part are specifically described in the copending applications Serial Nos. 463,814 and 463,815, filed October 29, 1942, the latter now abandoned, in the name of Winton I. Patnode and assigned to the same assignee as the present invention. The chain or branched chain compounds have the general formula $R_{(2a+2)}Si_aO_{(a-1)}$ where R represents the hydrocarbon radicals mentioned hereinbefore and a is a whole number equal to at least 9 for the purposes of the present invention.

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These compounds are further characterized by the fact that each oxygen atom is situated between two silicon atoms, each silicon atom is connected through oxygen to at least one other silicon atom and each terminal silicon atom is connected to three R groups. The cyclic compounds have the general formula $[R_2SiO]_n$ where R has the meaning hereinbefore indicated and n is a number equal to at least 9 for the purposes of the present invention. The cyclic compounds can be prepared by hydrolysis and condensation of a hydrolyzable silane of the formula R_2SiX_2 wherein R has the meaning hereinbefore indicated and X represents a hydrolyzable ester group, halogen atom, etc., followed by the separation of the lower boiling compounds containing less than about 9 silicon atoms per molecule. While the chain compounds can be similarly obtained by cohydrolysis of a suitable mixture of silanes of the formula $RSiX_3$ and R_2SiX_2 , with or without some R_3SiX , the preferred method of preparing these compounds comprises the treatment of a mixture of a polysiloxane of the formula $R_3Si-O-SiR_3$ and a cyclic polysiloxane having the unit structure $[R_2SiO]$ with sulphuric acid as described in the above-mentioned Pat-node application Serial No. 463,814. It will be noted that the liquid polysiloxanes, both chain compounds and cyclic compounds, employed in the practice of the present invention are characterized by an R-to-Si ratio of at least 2 and not more than about 2.22. In general, they will not contain more than about 16 or 17 silicon atoms per molecule.

The polysiloxanes containing 9 to 11 silicon atoms, preferably at least 10 silicon atoms, are most useful for low vacuum work, as in booster pumps. For example, in the operation of a small glass two-stage condensation pump with a methyl polysiloxane having the formula



which was 91% pure, the principal impurity being the cyclic compound $[(CH_3)_2SiO]_{10}$, a vacuum of 8×10^{-5} mm. was obtained at 30° C. A corresponding mixture of polysiloxanes containing 11 silicon atoms which was 85 per cent pure with regard to the chain compound, when tested in the same pump gave a vacuum of 2×10^{-5} mm. The pure straight chain compound containing nine silicon atoms has a vapor pressure of less than 3×10^{-4} mm. at room temperature. With the higher molecular weight polysiloxane fractions which usually consist of a mixture of both chain and cyclic compounds containing from 12 to 16 or 17 silicon atoms, vacua as low as 10^{-5} to 10^{-8} mm. can readily be obtained. These methyl polysiloxane compounds or mixtures thereof can be prepared by fractionating a mixture of chain, cyclic, or chain and cyclic methyl polysiloxanes to recover the fractions boiling between 140 and 225° C. at a pressure of from 1 to 10 microns.

The accompanying drawing shows a conventional oil-diffusion pump. This comprises a pump body 1, a central chimney 2 topped with a vapor deflector 3, and a heating element 4 which may be in the form of any convenient heat source. The liquid alkyl polysiloxane pump fluid is placed in the position designated in the drawing in an amount sufficient to cover the bottom of the chimney during operation. The pump sidearm is connected to a mechanical forepump which maintains a moderately low pressure in the pump body. The upper chamber is connected to the vessel to be evacuated. In operation, heat ap-

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plied to the pump liquid under the low pressure of the forepump causes the liquid alkyl polysiloxane to boil. The vapor rises through the chimney and discharges downward beneath the deflector, condensing on the outer walls and returning to the bottom. The vapor streaming down from the deflector catches air molecules from the space to be evacuated and drives them toward the mechanical forepump for removal.

In order to obtain a comparison between a polysiloxane fluid and the known organic materials as pumping fluids, a set of metal condensation pumps, as distinguished from the above-mentioned glass pump, were operated on continuous duty employing a methyl polysiloxane mixture containing more than 12 silicon atoms per molecule. These pumps had previously been operated with one of the best organic fluids (di-*n*-amyl sebacate). Between two and three months' operating time was about the maximum which could be obtained with this organic fluid since at the end of such period, the internal surfaces of the pumps were covered with tar and sludge while the fluid itself was contaminated with decomposition products. When charged and operated with the methyl polysiloxane fluid, the internal surfaces of these pumps and the operating fluid were still clean and bright when examined after five months' continuous use. It is thus evident that pumps operating with the alkyl polysiloxanes have the advantage of low maintenance costs due to freedom from costly periodic shutdowns for the purpose of cleaning the pump and changing the operating fluid. The fluids of the present invention also permit the operation of the condensation pump against higher back pressures since the marked heat-stability and oxidation-resistance of the alkyl polysiloxanes permit operation at higher boiler temperatures. Their high molecular weights which, in the case of the methyl compounds, range from about 660 for the 9 silicon compounds to over 1260 for the 17 silicon compounds and their correspondingly high molecular volumes, as compared with the organic fluids previously employed, permit the dimensions of the orifices of the condensation pumps and other parts of the pumps to be altered to permit operation with greater clearances resulting in a faster pumping action. It is to be understood, however, that the subject polysiloxanes can be employed in any condensation pump and that the present invention is not limited to pumps of any particular design or construction.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. Means for producing vacua which comprises a condensation pump containing a high molecular weight liquid alkyl polysiloxane as a pumping fluid, said polysiloxane containing from 9 to 17 silicon atoms per molecule.

2. Means for producing vacua which comprises a condensation pump containing a high-boiling liquid alkyl polysiloxane as a pumping fluid, said polysiloxane containing from 9 to 17 silicon atoms per molecule.

3. Means for producing vacua which comprises a condensation pump containing a liquid methyl polysiloxane as an operating fluid, the said polysiloxane containing at least nine and not more than 17 silicon atoms per molecule and having a methyl-to-silicon ratio of from 2.0 to 2.22.

4. Means for producing vacua which comprises a condensation pump containing a liquid methyl polysiloxane composition as the operating fluid, the said composition consisting essentially of

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methyl polysiloxane chain compounds containing at least nine and not more than 17 silicon atoms per molecule and having an average methyl-to-silicon ratio of from 2.0 to 2.22.

5. Means for producing vacua which comprises a condensation pump and, as an operating fluid for said pump, a liquid mixture consisting essentially of a major portion of methyl polysiloxane chain compounds and of cyclic methyl polysiloxane compounds both of which contain at least ten and not more than 17 silicon atoms and wherein the average methyl-to-silicon ratio of the said polysiloxane is from 2.0 to 2.22.

6. A means for evacuating closed vessels which comprises a condensation pump containing as the evacuating medium a liquid alkyl polysiloxane containing at least 9 and no more than 17 silicon atoms wherein the average alkyl-to-silicon ratio is from 2.0 to 2.22.

7. A high molecular weight condensation pump liquid adapted especially for use in producing vacua, the said liquid consisting essentially of a stable liquid alkyl polysiloxane having a ratio of from 2.0 to 2.22 alkyl groups per silicon atom and containing from 9 to 17 silicon atoms per molecule.

8. A high molecular weight liquid methyl polysiloxane condensation pump liquid adapted especially for use in producing vacua, the said liquid

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polysiloxane having a ratio of from 2.0 to 2.22 methyl groups per silicon atom, containing from 9 to 17 silicon atoms per molecule, and boiling between 140° to 225° C. at a pressure of from 1 to 10 microns.

9. A high molecular weight condensation pump fluid adapted especially for use in producing vacua, the said fluid consisting essentially of a mixture of liquid methyl polysiloxanes containing from 9 to 17 silicon atoms and having a methyl-to-silicon ratio of from 2.0 to 2.22, the molecular weight of the said polysiloxanes ranging from about 660 to 1260.

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