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(54) Title: METHOD OF REMOVING A VOLATILE ORGANOPOLYSILOXANE FROM A MIXTURE COMPRISING AN INERT GAS AND THE VOLATILE ORGANOPOLYSILOXANE

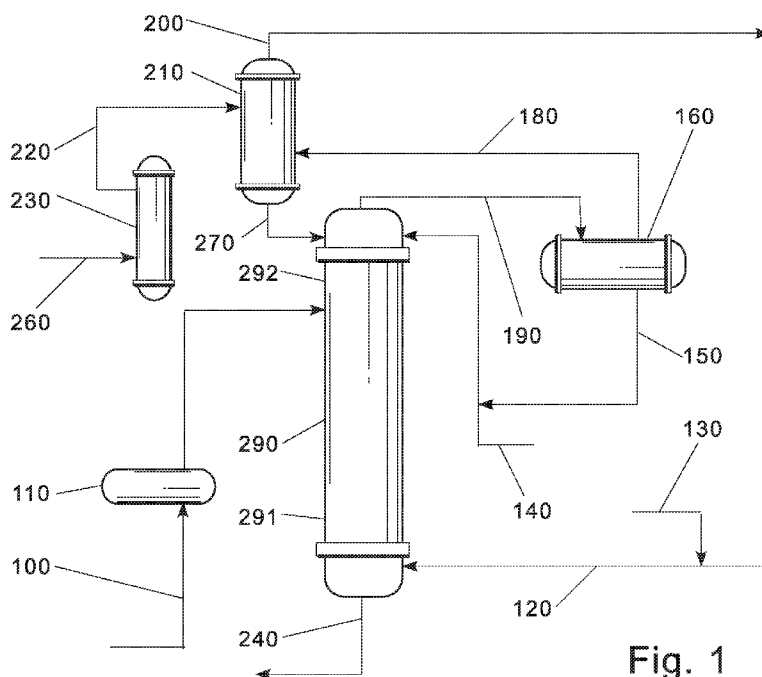


Fig. 1

(57) Abstract: A method for removing volatile organopolysiloxanes from a gaseous mixture comprising the volatile organopolysiloxane and an inert gas, the method comprising: contacting the gaseous mixture with a cool organopolysiloxane liquid to form a purified gas stream comprising the inert gas.

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METHOD OF REMOVING A VOLATILE ORGANOPOLYSILOXANE FROM A MIXTURE
COMPRISING AN INERT GAS AND THE VOLATILE ORGANOPOLYSILOXANE

CROSS-REFERENCE TO RELATED APPLICATIONS

5 [0001] None

FIELD OF THE INVENTION

[0002] The present invention relates, generally, to a method for removing volatile organopolysiloxanes from a gaseous mixture comprising the volatile organopolysiloxane and an inert gas, the method comprising: contacting the gaseous mixture with a cool
10 organopolysiloxane liquid to form a purified gas stream comprising the inert gas. The invention also relates to a method of removing volatile organopolysiloxanes from a mixture of comprising a volatile organopolysiloxane and non-volatile organopolysiloxane comprising contacting the mixture with an inert gas to form a first stream comprising non-volatile organopolysiloxanes and a second stream comprising a gaseous mixture comprising the
15 inert gas and the volatile organopolysiloxane.

BACKGROUND OF THE INVENTION

[0003] Manufacturers of organopolysiloxanes include a specification for maximum content of volatile material for siloxane products because volatile materials can cause performance issues in end uses for the siloxane products. In addition, potential regulations may also
20 require reduction of some specific volatile organopolysiloxanes, such as some cyclic organopolysiloxanes, in the future. Therefore, processes to purify siloxanes are important to the production of siloxanes, but the removal of volatile materials from low molecular weight siloxanes has been a challenge in siloxane production. In particular, the removal of volatile cyclic organopolysiloxanes from low molecular weight, reactive and non-reactive, non-cyclic
25 organopolysiloxanes has been a challenge for manufacturers.

[0004] Various methods for removing cyclic or volatile organopolysiloxanes from mixtures of organopolysiloxanes using various gases have been reported. For example, the use of steam to remove volatile cyclic siloxanes from short chain, silanol-terminated siloxanes by stripping in a distillation column has been disclosed. In addition, removing low molecular
30 weight siloxanes by bringing a multiplicity of jets of the siloxane into contact with gaseous carbon dioxide has been disclosed. In this particular method, carbon dioxide dissolves in the purified siloxane and creates layers of purified siloxane and unpurified siloxane that then must be separated, and the lower purity siloxane is then recirculated in the process to increase its purity. The entrained carbon dioxide is removed from the purified siloxane to

give the purified product. The carbon dioxide is either released into the atmosphere or recirculated in the process after condensation of the entrained volatiles. Further, a process for removing cyclic siloxanes from a siloxane emulsion has been disclosed in which cyclic siloxanes are stripped out of the emulsion using a falling film evaporator.

5 [0005] Even though processes have been disclosed to remove volatile materials, including cyclic siloxanes, from siloxanes, these processes still can be improved. For example, the processes described may operate at high temperatures. These high temperatures make the processes more energy and capital intensive, create more byproducts, and can increase chain lengths of non-cyclic polysiloxanes. Further, the processes may not efficiently remove
10 the volatile organopolysiloxane from the non-volatile organopolysiloxanes requiring recirculation of the organopolysiloxane mixture in the process to reduce the volatile cyclic organopolysiloxanes adequately which increases the time and energy required in the process. Still further, the processes using steam may add unwanted water or moisture to the final product. Finally, processes to remove the volatile cyclic organopolysiloxanes from a gas
15 once the gas has been used in removing the cyclic organopolysiloxanes from the organopolysiloxane mixture are required so that the gas may be recycled in the process. The gas should be efficiently reduced in volatile organopolysiloxane so that the gas can be efficiently used again in the process for removing volatile organopolysiloxane from an organopolysiloxane mixture.

20 [0006] Thus, a need still exists for processes to remove volatile organopolysiloxanes from non-volatile non-reactive, and reactive, organopolysiloxanes, such as silanol end-blocked organopolysiloxanes, with improved efficiency and that are conducted at lower temperatures to avoid the associated chain lengthening, byproducts, and energy requirements, and that do not introduce unwanted materials, such as moisture, into the organopolysiloxane.

25 Furthermore, there exists a need for a process to remove volatile organopolysiloxanes from a gaseous mixture comprising an inert gas and the volatile organopolysiloxanes that has good efficiency, and that reduces the amount of volatile organopolysiloxanes in the gas sufficiently for efficient use, or reuse, in processes to remove volatile organopolysiloxanes from non-volatile organopolysiloxanes.

30 BRIEF SUMMARY OF THE INVENTION

[0007] The present invention is directed to a method for removing a volatile organopolysiloxane from a gaseous mixture comprising the volatile organopolysiloxane and an inert gas, the method comprising: contacting the gaseous mixture with a cool organopolysiloxane liquid to form a purified gas stream comprising the inert gas.

[0008] The present invention is further directed to a method for removing a volatile organopolysiloxane from an organopolysiloxane mixture, the method comprising:

i) contacting an organopolysiloxane mixture comprising a volatile organopolysiloxane and a non-volatile organopolysiloxane with an inert gas to form a first stream comprising a gaseous mixture comprising the inert gas and the volatile organopolysiloxane and to form a second stream comprising the non-volatile organopolysiloxane;

ii) optionally removing a portion of the volatile organopolysiloxane from the first stream to form a partially purified first stream comprising the inert gas and the residual volatile organopolysiloxane and a third stream comprising the portion of the volatile organopolysiloxane removed from the first stream; and

iii) contacting the first stream from i) or, when ii) is present, the first stream from i) or the partially purified first stream from ii) with a cool organopolysiloxane liquid to form a purified gas stream and a volatile-containing cool organopolysiloxane stream comprising the cool organopolysiloxane and the volatile organopolysiloxane;

iv) optionally contacting the purified inert gas stream from iii) with the organopolysiloxane mixture in i), where the purified inert gas steam forms from greater than 0% to 100% of the gas contacted in i);

v) optionally cooling a portion of the second stream from i) to form the cool organopolysiloxane liquid in iii) where the cooled portion of the second stream from i) forms from > 0% to 100% of the cool organopolysiloxane in iii);

vi) optionally contacting the volatile-containing cool organopolysiloxane stream from iii) with the inert gas in i);

vii) optionally when ii) is present, contacting the third stream from ii) with the inert gas in i).

[0009] The method of the invention for removing volatile organopolysiloxanes from a gaseous mixture efficiently provides a purified inert gas stream that may be used, or reused, in processes for removing volatile organopolysiloxanes from low viscosity, non-volatile organopolysiloxanes.

[0010] The method of the invention further provides for the removal of volatile organopolysiloxanes from non-volatile organopolysiloxanes with good efficiency. The method allows for the quick separation of the volatile from the non-volatile organopolysiloxane so that the recycling of the non-volatile organopolysiloxane in the process to reduce the amount of volatile organopolysiloxanes in the non-volatile siloxane to

acceptable levels is reduced or eliminated, and the chain length of the non-volatile organopolysiloxane is not substantially increased in the process.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011] **Figure 1** is a schematic representation of one embodiment of the present process.

DETAILED DESCRIPTION OF THE INVENTION

[0012] The Brief Summary and Abstract are incorporated here by reference. The invention embodiments, uses and advantages summarized above are further described below.

[0013] Aspects of the invention are described herein using various common conventions.

For example, all states of matter are determined at 25 °C and 101.3 kPa unless indicated otherwise. All % are by weight unless otherwise noted or indicated. All % values are, unless otherwise noted, based on total amount of all ingredients used to make the composition, which adds up to 100 %. Any Markush group comprising a genus and subgenus therein includes the subgenus in the genus, e.g., in "R is hydrocarbyl or alkenyl," R may be alkenyl, alternatively R may be hydrocarbyl, which includes, among other subgenuses, alkenyl.

[0014] Aspects of the invention are described herein using various patent terms. For example, "alternatively" indicates a different and distinct embodiment. "Comparative" as used in comparative example, comparative process or comparative method means a non-invention experiment and should not be interpreted as prior art. "Comprises" and its variants (comprising, comprised of) are open ended. "Consists of" and its variants (consisting of) are closed ended. "Contacting" means bringing into physical contact. "May" confers a choice, not an imperative. "Optionally" means is absent, alternatively is present.

[0015] For illustration, the invention is described with reference to an embodiment of the invention as depicted in Figure 1; however, the invention is not limited to this one embodiment. One skilled in the art will recognize that the invention can be practiced using a number of slight variations of the method not described herein, and these variations are within the scope of the invention.

[0016] A first method for removing a volatile organopolysiloxane from a gaseous mixture comprising the volatile organopolysiloxane and an inert gas, the method comprising: contacting the gaseous mixture with a cool organopolysiloxane liquid to form a purified gas stream comprising the inert gas.

[0017] The gaseous mixture, the volatile organopolysiloxane, the cool organopolysiloxane liquid, the inert gas and the purified gas stream are as described for the second method below.

[0018] A second method for removing a volatile organopolysiloxane from an organopolysiloxane mixture, the method comprising:

i) contacting an organopolysiloxane mixture comprising a volatile organopolysiloxane and a non-volatile organopolysiloxane with an inert gas to form a first stream comprising a gaseous mixture comprising the inert gas and the volatile organopolysiloxane and to form a second stream comprising the non-volatile organopolysiloxane;

ii) optionally removing a portion of the volatile organopolysiloxane from the first stream to form a partially purified first stream comprising the inert gas and the residual volatile organopolysiloxane and a third stream comprising the portion of the volatile organopolysiloxane removed from the first stream; and

iii) contacting the first stream from i) or, when ii) is present, the first stream from i) or the partially purified first stream from ii) with a cool organopolysiloxane liquid to form a purified gas stream and a volatile-containing cool organopolysiloxane stream comprising the cool organopolysiloxane and the volatile organopolysiloxane;

iv) optionally contacting the purified inert gas stream from iii) with the organopolysiloxane mixture in i), where the purified inert gas steam forms from greater than 0% to 100% of the gas contacted in i);

v) optionally cooling a portion of the second stream from i) to form the cool organopolysiloxane liquid in iii) where the cooled portion of the second stream from i) forms from > 0% to 100% of the cool organopolysiloxane in iii);

vi) optionally contacting the volatile-containing cool organopolysiloxane stream from iii) with the inert gas in i);

vii) optionally when ii) is present, contacting the third stream from ii) with the inert gas in i).

[0019] To ease in understanding, the invention is described with reference to the embodiment of the invention depicted in Figure 1; however, the invention is not limited to this one embodiment. One skilled in the art will recognize that the invention can be practiced using a number of slight variations of the method not described herein, and these variations are within the scope of the invention.

[0020] With reference to Figure 1, inert gas **120** and an organopolysiloxane mixture **100** comprising a volatile organopolysiloxane and a non-volatile organopolysiloxanes are contacted in i) to form a first stream **190** comprising the inert gas and the volatile organopolysiloxane and a second stream **240** comprising the non-volatile

organopolysiloxane, wherein the organopolysiloxane mixture is at a temperature from 140 °C to 160 °C.

[0021] The inert gas is a gas such as nitrogen, argon, steam, or carbon dioxide. As used herein, "inert gas" is intended to mean a gas that will not react with the organopolysiloxane used in the process. In one embodiment, the inert gas is nitrogen. Nitrogen and other inert gases that may be used in the invention are available commercially. One skilled in the art would know how to obtain and use inert gases in the process of the invention.

[0022] The organopolysiloxane mixture comprises a volatile organopolysiloxane and a non-volatile organopolysiloxane, alternatively a volatile organopolysiloxane, a non-volatile organopolysiloxane, and an azeotroping solvent.

[0023] As used in reference to an organopolysiloxane, "volatile" means having a boiling point at or below 220 °C, alternatively below 215 °C, alternatively below 200 °C, alternatively below 150 °C, alternatively from 120 to 220 °C, alternatively from 120 to 215 °C.

[0024] In one embodiment, the volatile organopolysiloxane comprises 3 to 10, alternatively 3 to 8, alternatively 4 to 5 silicon atoms in a polysiloxane ring structure and has monomer units of the formula $-R_2SiO_{2/2}-$, wherein each R is independently C_{1-10} hydrocarbyl, alternatively C_{1-4} hydrocarbyl, alternatively R is C_{1-4} alkyl, alternatively methyl. Examples of hydrocarbyl groups represented by R include, but are not limited to, methyl, ethyl, propyl, 1-methylethyl, butyl, 1-methylpropyl, 2-methylpropyl, 1,1-dimethylethyl, pentyl, 1-methylbutyl, 1-ethylpropyl, 2-methylbutyl, 3-methylbutyl, 1,2-dimethylpropyl, 2,2-dimethylpropyl, hexyl, heptyl, octyl, nonyl, and decyl; cycloalkyl, such as cyclopentyl, cyclohexyl, and methylcyclohexyl; aryl, such as phenyl and naphthyl; alkaryl such as tolyl and xylyl; aralkyl, such as benzyl and phenylethyl; alkenyl, such as vinyl, allyl, and propenyl, butenyl, hexenyl, and octenyl; arylalkenyl, such as styryl and cinnamyl; and alkynyl, such as ethynyl and propynyl. Examples of cyclic organopolysiloxanes include, but are not limited to, hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane.

[0025] The volatile organopolysiloxane can be a mixture. In one embodiment, the volatile organopolysiloxane is a mixture comprising cyclic organopolysiloxanes. Examples of mixtures of cyclic organopolysiloxanes include, but are not limited to, a mixture comprising at least two of hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, and decamethylcyclopentasiloxane, alternatively a mixture comprising octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane.

[0026] The non-volatile organopolysiloxane may be a linear, branched, or a mixture of linear and branched organopolysiloxanes, alternatively a linear, branched, or mixture of linear or branched silanol end-capped organopolysiloxanes. As used herein, "silanol end-capped" means that the organopolysiloxane has an SiOH group on at least one terminus, alternatively two or more termini, of an organopolysiloxane chain.

[0027] As used in reference to the organopolysiloxane, "non-volatile" means having a boiling point above 215 °C, alternatively above 220 °C, alternatively above 225 °C, alternatively above 250 °C.

[0028] In one embodiment, the non-volatile organopolysiloxane in the organopolysiloxane mixture has a viscosity below 1000 cSt, alternatively below 100 cSt, alternatively below 50 cSt. Viscosity is measured at 25 °C using a Ubbelohde glass capillary, gravity-flow viscometer according to ASTM D-445, IP 71, where a clean, dry calibrated viscometer that will yield a flow time of greater than 80 s with the sample is used. The viscometer is loaded in the correct manner as required by the viscometer. The tube is placed in a constant temperature bath and allowed to reach equilibrium (at least 15 min. at 25 °C). Suction or pressure is used to adjust the head level of the sample to a point about 5 mm above the first timing mark. The sample is allowed to flow freely, and the timer is started as the meniscus passes the first timing mark. The timer is stopped as the meniscus passes the second timing mark, and the time is recorded to the nearest 0.1 s. The test is repeated without refilling the tube, and the average is used in calculating the viscosity. The viscometer is calibrated using either primary or secondary standards.

[0029] In one embodiment, the non-volatile organopolysiloxane in the organopolysiloxane mixture comprises one or more of the polymer units according to the formula $-R_2SiO_{2/2}$, $-R_3SiO_{1/2}$, $-R_2(OH)SiO_{1/2}$, $-RSiO_{3/2}$, and $SiO_{4/2}$, alternatively $-R_2SiO_{2/2}$, $-R_3SiO_{1/2}$, $-RSiO_{3/2}$, and $R_2(OH)SiO_{1/2}$, alternatively $-R_2SiO_{2/2}$, and $-R_2(OH)SiO_{1/2}$, where R is as defined above.

[0030] Examples of non-volatile organopolysiloxanes include, but are not limited to, linear and branched silanol end-capped polydimethylsiloxane, tetrakis-(trimethylsilyl)siloxane, tetrakis-(dimethylsiloxy)siloxane, tris-(trimethylsilyl)siloxane, tris-(dimethylsiloxy)siloxane, tris-(trimethylsilyl)(hydroxy)siloxane, and tris-(dimethylsiloxy)(hydroxyl)siloxane.

[0031] The non-volatile organopolysiloxanes may be a mixture of non-volatile organopolysiloxanes of varying viscosity, molecular weight and structure that give an overall viscosity and/or boiling point as described above. In one embodiment, the non-volatile organopolysiloxane comprises cyclic and non-cyclic structures, alternatively cyclic structures

having more than ten silicon atoms in the ring structure and non-cyclic structures, alternatively non-cyclic structures.

[0032] The organopolysiloxane mixture may comprise an azeotroping solvent. As used herein "azeotroping solvent" is intended to mean a substance added to an
5 organopolysiloxane mixture in order to form an azeotropic mixture for azeotropic distillation. Azeotropic and azeotropic-like mixtures are known in the art. For example, the following patents, the descriptions of which are all incorporated herein by reference, describe azeotropic mixtures: U.S. Pat. Nos. 5,492,647A; 5,478,493A; 5,454,970A; 5,454,972.

Azeotropic mixtures are mixtures of the components that vaporizes with no change in the
10 composition of the vapor from the liquid. Specifically, azeotropic mixtures boil without changing composition and evaporate at a temperature below their boiling point without changing composition. Accordingly, an azeotropic mixture may include mixtures of two components over a range of proportions where each specific proportion of the two components is an azeotropic mixture at a certain temperature but not necessarily at other
15 temperatures. The vapor pressure of low boiling azeotropic mixtures is higher, and the boiling point is lower, than the individual components. In fact, the azeotropic mixture has the lowest boiling point of any composition of its components. Thus, an azeotropic mixture can be obtained by distillation of a mixture whose composition initially departs from that of the azeotropic mixture.

[0033] An azeotropic-like mixture is a mixture of two components that acts like an azeotropic mixture. Thus azeotropic-like mixtures have constant boiling characteristics, or have a tendency not to fractionate upon boiling or evaporation. In an azeotropic-like mixture, the composition of the vapor formed during boiling or evaporation is identical or substantially
20 identical to the composition of the original liquid. During boiling or evaporation, the liquid changes only minimally, or to a negligible extent, if it changes at all. In other words, it has about the same composition in vapor phase as in liquid phase when employed at reflux. In contrast, the liquid composition of non-azeotropic-like mixtures change to a substantial degree during boiling or evaporation. By definition, azeotropic-like mixtures include all ratios of the azeotropic mixtures boiling within one °C of the minimum boiling point at 760 Torr.

[0034] An azeotroping solvent according to our invention is a material that forms an azeotropic mixture or azeotropic-like mixture with all or a portion of the volatile organopolysiloxanes or the non-volatile organopolysiloxanes in the organopolysiloxane mixture, alternatively with the volatile organopolysiloxane, alternatively with octamethylcyclotetrasiloxane in the organopolysiloxane mixture.
30

[0035] The azeotroping solvent is at least one solvent selected from the group consisting of a) n-butyl lactate, b) ethyl lactate, c) isopropyl lactate, d) 1-butoxy-2-ethanol, e) 1-methoxy-2-propanol, f) n-propoxypropanol, g) 1-butoxy-2-propanol, h) 2-pentanol, 2-methyl-1-pentanol, i) 3-methyl-3-pentanol, j) 1-hexanol, k) cyclohexanol, l) 2-methylcyclohexanol, m) 4-methylcyclohexanol, n) 1-heptanol, and o) a mixture of any two or more of a) through n). The azeotroping solvents of the invention are available commercially.

[0036] The amount of volatile organopolysiloxane in the organopolysiloxane mixture can vary. Typically, the organopolysiloxane mixture comprises up to 50% (w/w), alternatively up to 25% (w/w), alternatively up to 15% (w/w), alternatively from .01% ppmw to 50% (w/w), alternatively from 0.01% to 25% (w/w), alternatively from 0.10% to 25%, alternatively from 1.0% to 15% (w/w), based on the weight of the volatile organopolysiloxane and non-volatile organopolysiloxane, of volatile organopolysiloxane. Once skilled in the art would know how to determine the amount of volatile organopolysiloxane in the organopolysiloxane mixture.

[0037] The amount of non-volatile organopolysiloxane in the organopolysiloxane mixture can vary. The organopolysiloxane mixture comprises at least 50%, alternatively at least 75%, alternatively at least 85%, alternatively from 50 to 99.9999% (w/w) alternatively from 75 to 99.9999% (w/w), alternatively from 85 to 99.999% (w/w), alternatively 85 to 99.99% (w/w), based on the weight of the volatile organopolysiloxane and the non-volatile organopolysiloxane, of non-volatile organopolysiloxane.

[0038] Methods of making organopolysiloxane mixtures according to the invention are known in the art. For example, the organopolysiloxanes mixture may be made by the hydrolysis and condensation of organohalosilanes followed by partial separation by distillation. The organopolysiloxanes mixture may also be made by the rearrangement reaction of low molecular weight linear organopolysiloxanes. The azeotroping solvent may be added to and mixed with organopolysiloxanes in either a separate kettle or in-line as the organopolysiloxanes are transported to the equipment used for contacting with the inert gas. One skilled in the art would know how to make cyclic organopolysiloxanes, non-cyclic organopolysiloxanes, and the organopolysiloxane mixture according to the invention.

[0039] The first stream comprises the inert gas and a volatile organopolysiloxane.

Alternatively, the first stream comprises the inert gas, the volatile organopolysiloxane, and the non-volatile organopolysiloxane. The inert gas, the volatile organopolysiloxane, and the non-cyclic organopolysiloxane in the first stream in i) are as described above.

[0040] The first stream **190** in i) comprises up to 80% (w/w), alternatively up to 75% (w/w), alternatively from 50 to 75% (w/w), based on the weight of the inert gas and volatile and non-volatile organopolysiloxane, of the volatile organopolysiloxane.

5 [0041] The first stream **190** in i) comprises up to 10 %, alternatively from 0.5 to 7% (w/w), alternatively from 1 to 5,% (w/w), based on the weight of the inert gas and volatile and non-volatile organopolysiloxane, of non-volatile organopolysiloxane.

[0042] The second stream **240** comprises purified non-volatile organopolysiloxane. The chemical structure and viscosity of the purified non-volatile organopolysiloxane is as described above for the non-volatile organopolysiloxane in the organopolysiloxane mixture.

10 [0043] The second stream **240** may comprise some residual volatile organopolysiloxane. The concentration of the volatile organopolysiloxane in the second stream **240** is less than the volatile organopolysiloxane concentration in the organopolysiloxane mixture. The second stream may comprise less than 1%, alternatively less than 10,000 ppm, alternatively, less than 1,000 ppm, alternatively less than 100 ppm, based on the weight of the volatile and
15 non-volatile organopolysiloxane, of volatile organopolysiloxane. The chemical structure and viscosity of the volatile organopolysiloxane are as described above for the organopolysiloxane mixture.

[0044] The mole ratio of the inert gas **120** to the organopolysiloxane mixture **100** contacted in i) is from 0.05 to 0.5, alternatively from 0.05 to 0.25. One skilled in the art would know how
20 to adjust the inert gas and organopolysiloxane mixture flow rates in column **290** to achieve the ratios of inert gas to the organopolysiloxane mixture.

[0045] The inert gas and organopolysiloxane mixture are contacted in column **290**. The column **290** may comprise a stripping section **291** and may comprise a reflux section **292**. When both are present, the stripping section **291** is below the reflux section **292**. In one
25 embodiment, the column comprises both a stripping section **291** and a reflux section **292**. One skilled in the art would know what a column and a stripping and reflux sections of a column are. Columns are available commercially.

[0046] The inert gas **120** that enters the column **290** below the stripping section **291** and flows up through the stripping section **291** contacting the organopolysiloxane mixture **100**
30 and then, with the volatile organopolysiloxane removed from the organopolysiloxane mixture **100**, through the reflux section **292**, when present, before exiting the column in the first stream **190**. The first stream **190** exits the column near the top of the column. The inert gas **120** and organopolysiloxane mixture **100** flow countercurrent through stripping section **291** of column **290**.

[0047] The temperature of the inert gas **120** contacted in i) is not critical. The temperature of the inert gas may be from 20 °C to 150 °C, alternatively from 40 °C to 145 °C, alternatively from 120 °C to 140 °C. The temperature of the inert gas may be achieved by preheating the inert gas prior to contacting in i). One skilled in the art would know how to adjust the temperature of inert gas by, for example, sending the gas through a heater.

[0048] The organopolysiloxane mixture **100** may enter the column above the stripping section **291**, or, in one embodiment, above the stripping section and below the reflux section **292**, when present, of the column **290** and flows countercurrent to the inert gas **120** in the stripping section. The second stream **240** exits the column near the bottom of the stripping section **291** of the column **290**.

[0049] One skilled in the art would know how to achieve the flow of inert gas **120** and the organopolysiloxane mixture **100** through a column **290**. The process may be a continuous process, where the inert gas **120** and the organopolysiloxane mixture **100** are continually flowed countercurrently through the column **290**.

[0050] The temperature of the organopolysiloxane mixture **100** in i) may vary. The organopolysiloxane mixture **100** contacted in i) may have a temperature from 120 to 200 °C, alternatively from 130 to 180 °C, alternatively from 140 to 160 °C.

[0051] The temperature of the organopolysiloxane mixture in i) may be achieved by preheating the organopolysiloxane mixture prior to contacting with the inert gas using a heater **110**. One skilled in the art would know how to preheat the organopolysiloxane mixture and how to adjust the temperature of the organopolysiloxane mixture using a heater **110**. Any heater suitable for preheating the organopolysiloxane mixture of the invention may be used as the heater **110**.

[0052] The temperature of the stripping section **291** of the column may vary. Typically the temperature is from 115 to 175 °C, alternatively from 120 to 160 °C, alternatively from 125 to 150 °C. One skilled in the art would know how to vary the temperature in the stripping section of a column.

[0053] The temperature of the reflux section **292**, when present, of the column may vary. Typically the temperature of the reflux section **292** is from 80 to 190 °C, alternatively from 85 to 180 °C, alternatively from 90 to 150 °C. One skilled in the art would know how to vary the temperature in the reflux section of a column.

[0054] The pressure in the column **290** may be from atmospheric to below atmospheric pressure, alternatively from 0 to 27 kPa, alternatively from 10 to 17 kPa. One skilled in the art would know how to vary the pressure in the column **290**.

[0055] The water content of the second stream is less than or equal to 1000 micrograms/gram ($\mu\text{g/g}$), alternatively less than or equal to 850 $\mu\text{g/g}$, alternatively less than or equal to 800 $\mu\text{g/g}$, alternatively less than or equal to 500 $\mu\text{g/g}$, based on the weight of the entire second stream. The amount of water in the second stream can be measured by Karl Fisher titration as described in the examples.

[0056] A portion of the volatile organopolysiloxane may be removed from the first stream **190** in ii) to form a partially purified first stream **180** comprising the inert gas and a third stream **150** comprising the portion of the volatile organopolysiloxane removed from the first stream. The volatile organopolysiloxane may be removed from the first stream by condensing the volatile organopolysiloxane from the first stream **190** in a condenser **160**, alternatively the volatile organopolysiloxane may be removed using an absorber **210** as described below for iii). The volatile organopolysiloxane removed in ii) and the inert gas in the purified first stream in ii) are as described above for i). One skilled in the art would know how to use a condenser **160** to remove a portion of the volatile organopolysiloxane from the first stream **180**. Condensers are available commercially.

[0057] In iii), the first stream **190** from i), or, when ii) is present, the first stream **190** from i) or the partially purified first stream **180** from ii) are contacted with a cool organopolysiloxane comprising an organopolysiloxane, wherein the cool organopolysiloxane is at a temperature up to 60 °C, alternatively up to 40 °C, alternatively from 0 to 40 °C, alternatively from 20 to 40 °C, and the first steam from **190** or the partially purified first stream from **180** are at a temperature from 30 °C to 170 °C, alternatively from 40 °C to 160 °C, alternatively from 50 °C to 100 °C, in an absorber **210** to form a purified nitrogen stream **200** and a volatile-containing organopolysiloxane stream **270** comprising the cool organopolysiloxane and the volatile organopolysiloxane. One skilled in the art would know the meaning of an absorber. For example, the absorber may consist of a tank containing the cool organopolysiloxane where the first stream **190** or partially purified first stream **180** is either bubbled through the cool organopolysiloxane, flowed over the top of the cool organopolysiloxane, or flowed over a falling film of the cool organopolysiloxane. Absorbers are available commercially.

[0058] The cool organopolysiloxane comprises an organopolysiloxane. The organopolysiloxane in the cool organopolysiloxane is not limited except that it must be able to absorb the volatile organopolysiloxane from the first stream from i) or the partially purified stream from ii). In one embodiment, the organopolysiloxane comprised by the cool organopolysiloxane is as described for the non-volatile organopolysiloxane in the organopolysiloxane mixture above.

[0059] In one embodiment, the cool organopolysiloxane comprises from no detectable amounts to a small amount of the volatile organopolysiloxane. A small amount of volatile organopolysiloxane means an amount that will not negatively impact the absorption of the volatile organopolysiloxane from the first stream from i) or the partially purified stream from
5 ii), alternatively less than 1%, alternatively less than 10,000 ppm, alternatively, less than 1,000 ppm, alternatively less than 100 ppm, alternatively from 1 to 100 ppm, based on the weight of the volatile and non-volatile organopolysiloxane in the cool organopolysiloxane, of volatile organopolysiloxane.

[0060] The purified inert gas stream **200** in iii) may comprise the inert gas from the first
10 stream **190** or the partially purified first stream **180**. The purified inert gas stream **200** comprises less than 10,000, alternatively less than 1,000, alternatively less than 100 ppmw, based on the weight of the inert gas and any organopolysiloxane, of volatile organopolysiloxane.

[0061] The volatile-containing organopolysiloxane stream **270** in iii) may comprise the
15 volatile organopolysiloxane from the first stream **190** or the partially purified first stream **180** and the organopolysiloxane from the cool organopolysiloxane **220**. The inert gas and cyclic organopolysiloxane are as described for i) above.

[0062] The volatile-containing organopolysiloxane stream **270** from iii) comprises up to 1% w/w, alternatively up to 10,000 ppm, alternatively up to 1,000 ppm, alternatively up to 100
20 ppm, alternatively from 1 to 100 ppm cyclic organopolysiloxane. The volatile organopolysiloxane in the volatile-containing organopolysiloxane stream **270** from ii) is as described for i) above.

[0063] In iv), the purified inert gas stream **200** from iii) is contacted with the organopolysiloxane mixture **100** in i). The purified inert gas stream **200** may be from 0% to
25 100%, alternatively from greater than 0 to 100% of the inert gas contacted in i).

[0064] The amount of volatile organopolysiloxane in the purified inert gas stream **200** affects the efficiency of the removal of cyclic organopolysiloxane from the organopolysiloxane mixture with increasing amounts of volatile organopolysiloxane in the purified inert gas stream reducing the efficiency of the removal of cyclic organopolysiloxane
30 from the organopolysiloxane mixture. The purified inert gas stream **200** may have up to 10,000 ppmw, alternatively up to 7,000 ppmw, alternatively from 1 to 4,000, alternatively from 1 to 100 ppmw, based on the weight of the volatile organopolysiloxane and the inert gas, of cyclic organopolysiloxane.

[0065] A portion of the second stream **240** from i) may form stream **260** and cooled in v) using a chiller **230** to form the cool organopolysiloxane in iii). The cool organopolysiloxane may comprise from 0 to 100%, alternatively from greater than 0 to 100% of the second stream from i). Therefore, the cool organopolysiloxane in iii) may comprise the non-volatile organopolysiloxane or a mixture of volatile and non-volatile organopolysiloxanes. One skilled in the art would know how to use a chiller **230** to cool the second stream **240** or other organopolysiloxane stream **260** to the temperature of the cool organopolysiloxane in iii). Any chiller suitable for chilling organopolysiloxanes may be used.

[0066] In vi), the volatile-containing organopolysiloxane stream **270** may be contacted with the inert gas **120** in i). The volatile-containing organopolysiloxane stream **270** is contacted with the inert gas by introducing the volatile-containing organopolysiloxane stream **270** to the column **290** near the top of column **290** above the stripping section **291** and, in one embodiment, above of the reflux section **292**, when present.

[0067] In vii), when ii) is present, a portion of the third stream **150** from ii) may be contacted with the inert gas in i) by introducing the third stream to column **290** near the top of the column **290** above stripping section **291**, or, in one embodiment, above the reflux section **292**, when present. The volatile organopolysiloxane comprised by the third stream **150** and the process conditions for the contacting with the inert gas in i) are as described in i).

[0068] The method of the invention removes volatile organopolysiloxane from a mixture with non-volatile organopolysiloxane providing a purified non-volatile organopolysiloxane with low levels of volatile organopolysiloxane with good efficiency. The method allows for the quick separation so that the recycling of the non-volatile organopolysiloxane in the process to further reduce the amount of volatile organopolysiloxanes to acceptable levels is reduced, thereby decreasing the possibility of increasing chain lengths, viscosity, branching, silanol content, or other characteristics of the non-volatile organopolysiloxane as well as reducing the energy requirements of the process.

[0069] The volatile and non-volatile organopolysiloxanes separated in the present method can be sold commercially or used to make other molecular weight and structure polysiloxanes. For example, the volatile organopolysiloxanes may be polymerized to make linear organopolysiloxanes of particular chain lengths or equilibrated to make cyclic organopolysiloxanes of different ring sizes. The non-volatile organopolysiloxanes may be reacted to increase chain lengths or used as additives in formulations in various industries and/or products. These products are sold into many different end-use applications.

EXAMPLES

[0070] The invention is further illustrated by, and an invention embodiment may include any combinations of features and limitations of, the non-limiting examples thereof that follow.

[0071] The following examples are included to demonstrate particular embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples which follow represent techniques discovered by the inventor to function well in the practice of the invention, and thus can be considered to constitute modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention. Unless otherwise indicated, all percentages are in weight % (wt. %). When the unit 'parts per million' (ppm) are used in the example, it is based on weight. The following table describes the abbreviations used in the examples:

Table 2. List of abbreviations used in the examples.

Abbreviation	Word
g	gram
wt	weight
%	percent
hr	hour
°C	degrees Celsius
mL	milliliters

Test Methods

[0072] Water content was measured by Karl Fischer using a Metrohm 774 oven accessory and Metrohm 851 coulometric titrator. Samples were heated to 130 °C during analysis.

[0073] The quantities and identities of polysiloxanes were determined by analyses using a gas chromatograph equipped with capillary column, flame ionization detector and a headspace autosampler. The system was calibrated by analyzing known standards of polysiloxanes relative to an internal standard (mesitylene). Sample preparation included the use of hexamethyldisilazane to serve as an endcapping agent for the hydroxy terminated linear species.

[0074] The base experiments in Examples 1-3 were based on Aspen models of the system.

Example 1

[0075] OH-endblocked dimethyl silicone fluid (Dow Corning 4-2737 Fluid) was fed into a 12-tray glass column at 151 °C and 5.6 g/min. The starting material had %OH of 4.12, 1.1% D4, and 1.3% D5. Nitrogen was fed in countercurrent at 310 standard cubic centimeters per minute into the system, and the system was operated at a pressure of 100 mmHg. The final product had 4.17% OH, a non-detectable level of D4, and 0.12% D5.

Example 2

[0076] OH-endblocked dimethyl silicone fluid (Dow Corning 4-2737 Fluid) was fed into a 12-tray glass column at 145 °C and 4.5 g/min. The starting material had %OH of 4.2, 1.15% D4, and 1.26% D5. Nitrogen was fed in countercurrent at 680 standard cubic centimeters per minute into the system, and the system was operated at a pressure of 100 mmHg. The final product had 4.24% OH, 0.06% D4 (octamethylcyclotetrasiloxane), and a non-detectable level of D5 (decamethylcyclopentasiloxane). This experiment was conducted with a nitrogen recycle. The nitrogen was passed through a water-cooled condenser, returned to atmospheric pressure using a vacuum pump, and recycled to the system.

Example 3

[0077] OH-endblocked dimethyl silicone fluid (JS-209) was fed into a 12-tray glass column at 154 °C and 2.3 g/min. The starting material had %OH of 7.4, 7.0% D4, and 4.7% D5. Nitrogen was fed in countercurrent at 496 standard cubic centimeters per minute into the system, and the system was operated at a pressure of 100 mmHg. The final product had 7.54% OH, 0.03% D4, and 0.04% D5.

[0078] This experiment was conducted with a nitrogen recycle. The nitrogen was passed through a water-cooled condenser, returned to atmospheric pressure using a vacuum pump, and recycled to the system.

Example 4

[0079] An organopolysiloxane mixture (of 1.55% octamethylcyclotetrasiloxane, 1.49% decamethylcyclopentasiloxane, 0.82% dodecamethylcyclohexasiloxane, and the remainder consisting of a mixture of higher molecular weight linear and cyclic organopolysiloxanes) comprising a volatile organopolysiloxane and a non-volatile organopolysiloxane (4.9% volatile and 95.1% non-volatile components) at a temperature of 155 °C was contacted with nitrogen gas in a column at an overhead pressure of 95 mmHg and overhead temperature of 87.8 °C and an organopolysiloxane mixture to nitrogen feed ration of 9.7:1 to form a first stream comprising a gaseous mixture of nitrogen and the volatile organopolysiloxane (56.1% volatile components and 43.9% nitrogen) and a second stream comprising the non-volatile

organopolysiloxane (0.045% octamethylcyclotetrasiloxane, 0.006%
decamethylcyclopentasiloxane, 0.097% dodecamethylcyclohexasiloxane and a mixture of
higher molecular weight linear and cyclic organopolysiloxanes) with less than a 15% change
in viscosity and less than 3% change in silanol content between the original mixture and
5 second stream. The second stream contained 800 µg/g, based on the entire weight of the
second stream.

[0080] A portion of the volatile organopolysiloxane from the first stream was removed
using a condenser to form a partially purified first stream comprising the nitrogen and the
residual volatile organopolysiloxane and a third stream comprising the portion of the volatile
10 organopolysiloxane removed from the first stream at a pressure of 95 mmHg and
temperature of 43.3 °C, with the third stream comprised of <5% water, <5%
hexamethylcyclotrisiloxane, 30.5% octamethylcyclotetrasiloxane, 29.5%
decamethylcyclopentasiloxane, 14.3% dodecamethylcyclohexasiloxane, and the remainder
comprised of other higher molecular weight linear and cyclic organopolysiloxanes.

[0081] The partially purified first stream comprising the nitrogen and residual volatile
organopolysiloxane was then contacted, at 91.5 mmHg, with a cool organopolysiloxane
liquid (comprising less than 0.2% (w/w) octamethylcyclotetrasiloxane,
decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane, and the remainder a
mixture of higher molecular weight linear and cyclic organopolysiloxanes and at 31 °C) to
20 form a purified gas stream and a volatile-containing cool organopolysiloxane stream
comprising the cool organopolysiloxane and the volatile organopolysiloxane. The volatile-
containing cool organopolysiloxane stream comprised about 17%
octamethylcyclotetrasiloxane, decamethylcyclotetrasiloxane, and
dodecamethylcyclohexasiloxane and the remainder comprised of other linear
25 organopolysiloxanes.

[0082] The purified nitrogen stream was then recycled and contacted with the
organopolysiloxane mixture, where the purified nitrogen steam formed 100% of the gas
contacted, at the conditions described above.

[0083] Approximately 8% of the second stream above was cooled to 31 °C to form the cool
30 organopolysiloxane liquid contacted with the partially purified first stream comprising
nitrogen. The volatile-containing cool organopolysiloxane stream was recycled and
contacted with the nitrogen in the column, and the third stream was recycled and contacted
with the nitrogen in the column at a mass ratio of 0.8:1 of the third stream to the inert gas.

That which is claimed is:

1. A method for removing volatile organopolysiloxanes from a gaseous mixture comprising the volatile organopolysiloxane and an inert gas, the method comprising:

5 contacting the gaseous mixture with a cool organopolysiloxane liquid to form a purified gas stream comprising the inert gas.

2. The method as in claim 1, wherein the cool organopolysiloxane liquid is at a temperature from 20 °C to 60 °C.

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3. The method as in any one of the preceding claims, wherein the cool organopolysiloxane comprises linear organopolysiloxanes.

4. The method as in any one of the preceding claims, wherein the gaseous mixture and the cool organopolysiloxane are contacted in an absorber and the pressure in the absorber is from 0 to 27 kPa, and wherein the gaseous mixture is at a temperature up to 200 °C.

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5. The method as in any one of the preceding claims, wherein the gas is nitrogen, argon, carbon dioxide or steam.

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6. The method as in any one of the preceding claims, wherein the volatile organopolysiloxane comprises hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, tetradecamethylcycloheptasiloxane, hexadecamethylcyclooctasiloxane, octadecamethylcyclononasiloxane, or eicosamethylcyclodecasiloxane.

25

7. A method for removing a volatile organopolysiloxane from an organopolysiloxane mixture, the method comprising:

i) contacting an organopolysiloxane mixture comprising a volatile organopolysiloxane and a non-volatile organopolysiloxane with an inert gas to form a first stream comprising a gaseous mixture comprising the inert gas and the volatile organopolysiloxane and to form a second stream comprising the non-volatile organopolysiloxane;

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ii) optionally removing a portion of the volatile organopolysiloxane from the first stream to form a partially purified first stream comprising the inert gas and the residual volatile organopolysiloxane and a third stream comprising the portion of the volatile organopolysiloxane removed from the first stream; and

5 iii) contacting the first stream from i) or, when ii) is present, the first stream from i) or the partially purified first stream from ii) with a cool organopolysiloxane liquid according to the method of claim 1 to form a purified gas stream and a volatile-containing cool organopolysiloxane stream comprising the cool organopolysiloxane and the volatile organopolysiloxane;

10 iv) optionally contacting the purified inert gas stream from iii) with the organopolysiloxane mixture in i), where the purified inert gas stream forms from greater than 0% to 100% of the gas contacted in i);

15 v) optionally cooling a portion of the second stream from i) to form the cool organopolysiloxane liquid in iii) where the cooled portion of the second stream from i) forms from > 0% to 100% of the cool organopolysiloxane in iii);

 vi) optionally contacting the volatile-containing cool organopolysiloxane stream from iii) with the inert gas in i);

 vii) optionally when ii) is present, contacting the third stream from ii) with the inert gas in i).

20 8. The method of claim 7, wherein one or more of ii), iv), v), and vi) are present as in one of the following limitations a) through o):

 a) v and vi; b) ii and iv; c) ii and v; d) ii and vi; e) ii, iv and v; f) ii, iv and vi; g) ii, v, and vi; h) ii, iv, v and vi; i) iv and v; j) iv and vi; k) iv, v, and vi; l) ii; m) iv, n) v, or o) vi.

25 9. The method of claim 7 or 8, wherein the method includes a column positioned substantially vertically and having a reflux section above a stripping section, wherein the inert gas and the organopolysiloxane mixture in i) are contacted in the stripping section of the column by introducing the inert gas in i) into the column below the stripping section, and
30 introducing the organopolysiloxane mixture in i) into the column above the stripping section, wherein the inert gas and organopolysiloxane mixture flow countercurrently through the stripping section of the column, and wherein the first stream in i) exits the column above the reflux section, and the second stream in i) exits the column below the stripping section.

10. The method as in any one of claims 7-9, wherein when iv) is present, the purified inert gas stream from iii) is introduced into the column below the stripping section, wherein when vi) is present, the volatile-containing cool organopolysiloxane stream from iii) is introduced into the column at the top of the reflux section, and wherein when vii) is present, the third stream from ii) is introduced into the column at the top of the reflux section in i).

11. The method as in any one of the preceding claims, wherein the organopolysiloxane mixture further comprises an azeotroping solvent.

12. The method as in any one of claims 7-11, wherein the organopolysiloxane mixture in i) comprises up to about 15% (w/w) of the volatile organopolysiloxane, based on the weight of the volatile organopolysiloxane and the non-volatile organopolysiloxane.

13. The method as in any one of claims 7-12, wherein the temperature of the cool organopolysiloxanes is from 20 to 30 °C and the organopolysiloxane mixture in i) is at a temperature from 140 °C to 160 °C and has a viscosity up to and including 50 cSt.

14. The method as in any one of the preceding claims, wherein the process is a continuous process and the moisture content of the second stream is less than or equal to 1000 µg/g, based on the weight of the entire second stream.

15. The method of any one of claims 7-14, wherein the non-volatile organopolysiloxane in the organopolysiloxane mixture is silanol end-capped, the volatile organopolysiloxane is cyclic, the inert gas is nitrogen, and wherein the organopolysiloxane mixture is preheated before contacting with the gas in i).

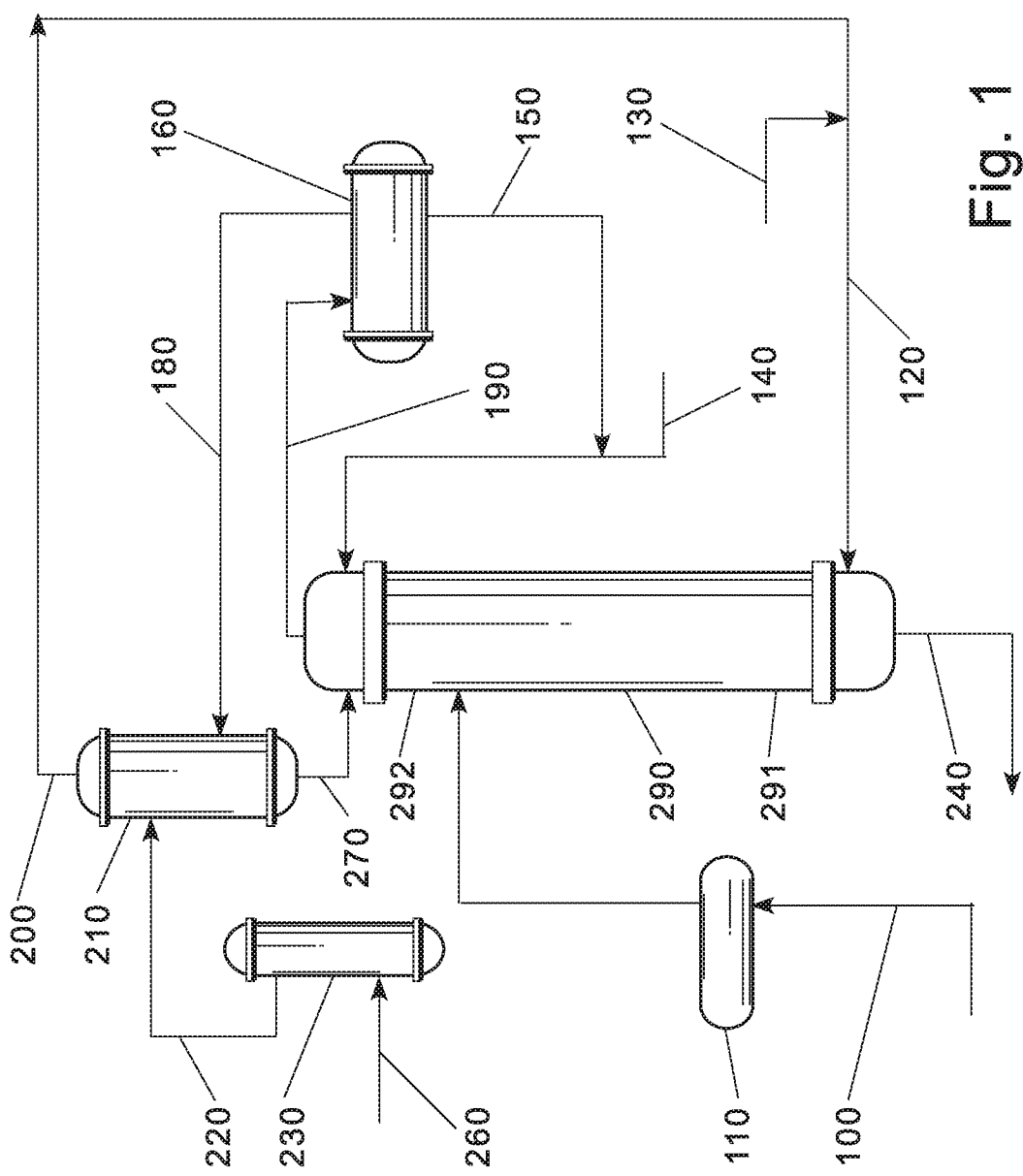


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2016/021923**A. CLASSIFICATION OF SUBJECT MATTER****C08G 77/34(2006.01)i, C08G 77/36(2006.01)i, C08G 77/04(2006.01)i**

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08G 77/34; C07F 7/08; C08G 77/00; C08G 77/36; C08G 77/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal) & Keywords: organopolysiloxane, cyclic organopolysiloxane, purification, cool organopolysiloxane, nitrogen gas, recirculation

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2834754 A (HATCH, R. L. et al.) 13 May 1958 See column 3, line 25 - column 6, line 3; and claim 7.	1-3,7-9
A	US 4895967 A (CRIVELLO, J. V. et al.) 23 January 1990 See claims 1-10.	1-3,7-9
A	US 3493595 A (STRASSER, R. et al.) 03 February 1970 See claims 1 and 2.	1-3,7-9
A	US 2006-0241266 A1 (GAMBUT-GAREL, L. et al.) 26 October 2006 See claims 18-34.	1-3,7-9
A	ACCETTOLA, F. et al., Siloxane removal from biogas by biofiltration: biodegradation studies, Clean Technologies and Environmental Policy, 2008, Vol. 10, No. 2, pp. 211-218 See abstract.	1-3,7-9



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2016/021923

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2834754 A	13/05/1958	None	
US 4895967 A	23/01/1990	EP 0361274 A2 EP 0361274 A3 EP 0361274 B1 JP 02-129192 A JP 06-086469 B2 KR 10-0152257 B1	04/04/1990 27/03/1991 08/06/1994 17/05/1990 02/11/1994 15/10/1998
US 3493595 A	03/02/1970	DE 1645671 A1 DE 1645671 B2 GB 1127097 A NL 153571 B NL 6708429 A	24/09/1970 13/09/1973 11/09/1968 15/06/1977 28/12/1967
US 2006-0241266 A1	26/10/2006	CN 101756791 A EP 1590396 A1 EP 1590396 B1 FR 2850660 A1 FR 2850660 B1 JP 2006-514153 A JP 4690201 B2 US 7750077 B2 WO 2004-072149 A1	05/04/2006 02/11/2005 03/12/2008 06/08/2004 04/08/2006 27/04/2006 01/06/2011 06/07/2010 26/08/2004