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AUSTRALIA

PATENTS ACT 1990

PATENT REQUEST: STANDARD PATENT

I/We, the Applicant(s)/Nominated Person(s) specified below, request I/We be granted a patent for the invention disclosed in the accompanying standard complete specification.

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[54] Invention Title:

Process and Apparatus for Generating a Vacuum

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By:

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NOTICE OF ENTITLEMENT

I, Albertus Wilhelmus Joannes ZEESTRATEN, of Carel van Bylandtlaen 30, 2596 HR The Hague, the Netherlands, being authorised by the Applicant/Nominated Person in respect of Application No. 29967/92 state the following:
The Applicant/Nominated Person has entitlement from the actual inventor(s) as follows:
The Applicant/Nominated Person is the assignee of the actual inventor(s).

The Applicant/Nominated Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s) lighted on the Person is the applicant of the basic application(s).

cation(s) listed on the Patent Request.

The basic application(s) listed on the Patent Request is/are the application(s) first made in a Convention Country in respect of the invention.

,J

DATED this 8th day of November , 199 2

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PROFILE ARGANISATION

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(56) Prior Art Documents
US 4198190
GB 1435942
AU 89079/91 F04F 5/46

(57) Claim

- 1. A process for the generation of a vacuum in a vessel comprising generating a vapour by heating a reservoir of a paraffin wax, discharging the vapour through an inlet into a gas entrainment zone connected to the vessel, thereby generating a vapour jet in the entrainment zone, allowing gas from the vessel to enter the vapour jet, allowing the vapour jet containing the gas to pass to a condensation zone, condensing vapour from the vapour jet in the condensation zone and separately recovering the condensed wax and the uncondensed material.
- 11. A process for the distillation of a paraffin wax comprising generating a vapour by heating a reservoir of a paraffin wax as defined in any one of claims 1 to 9, discharging the vapour through an inlet into a gas entrainment zone, thereby generating a vapour jet in the entrainment zone, evaporating the wax to be distilled in a distillation zone, allowing at least a part of the evaporated wax from the distillation zone to contact the vapour jet in the entrainment zone, allowing the vapour jet containing the evaporated wax to pass to a condensation zone, condensing vapour from the vapour jet containing the evaporated wax in the condensation zone and separately recovering the condensed wax and the uncondensed material.

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COMPLETE SPECIFICATION

FOR A STANDARD PATENT

ORIGINAL

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Invention Title:

Process and Apparatus for Generating a Vacuum

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

PROCESS AND APPARATUS FOR GENERATING A VACUUM

The present invention relates to a process for the preparation of a vacuum, to pumping apparatus, in particular to a diffusion pump, for use in the process, to fluids for use as pump fluids in the process and to applications of the process, in particular the refining of heat sensitive materials.

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Many industrial operations rely for their effectiveness on the generation and maintenance of a vacuum within an apparatus. Examples of such process include vacuum distillation, necessary for the refining of heat sensitive materials, and freeze-drying as applied, for example, in the food industry.

The process and apparatus employed in generating the vacuum depends upon the degree of vacuum required, which is in turn is determined by the particular operation concerned. Thus, rough vacua, ranging in absolute pressure from about 10² to about 10⁵ Pa, and medium vacua, ranging in pressure from about 10⁻¹ to about 10² Pa, may conveniently be generated using displacement pumps, for example liquid and gas ring vacuum pumps, rotary vane, rotary piston and rotary lobe vacuum pumps. To achieve the lower pressures typical of high vacua, ranging from 10⁻⁵ to 10⁻¹ Pa, and ultra high vacua, having pressures below 10⁻⁵ Pa, kinetic vacuum pumps, such as jet pumps, diffusion pumps and drag vacuum pumps, or entrapment vacuum pumps, such as sorption pumps and condensation pumps, are applied.

In the refining of many heat sensitive materials, vacuum distillation is applied. Typical apparatus for carrying out the vacuum distillation includes falling film and wiped film evaporators. Such processes are typically operated under vacuum of pressures of from 10^{-1} to 10^2 Pa, the generation of which is conveniently achieved using a diffusion vacuum pump.

In operation of the diffusion vacuum pump, a reservoir of pump fluid is heated to vaporise the fluid, which vapour then issues

from an aperture in a nozzle forming a high speed vapour jet. The gases being pumped diffuse into and are entrained in the vapour jet and are thereby carried by the jet to a region remote from the nozzle where the pump fluid vapour is condensed, separated from the gases being pumped and recycled to the reservoir. The gases being pumped, once separated from the pump fluid, are removed from the region by a pump, commonly referred to as a "backing pump" or "forepump". For a description of many aspects of vacuum technology in general and diffusion vacuum pumps in particular, reference is made to Ullmann's Encyclopaedia of Industrial Chemistry, Volume B 3, Unit Operations II, Section 21. For descriptions of specific embodiments of the diffusion vacuum pump, reference is made to US Patent Specifications Numbers 2,028,340, 2,338,583, 2,358,067 and 2,560,913 and to West German Patent Specification Number 883,185.

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Fluids for use as pump fluids in diffusion vacuum pumps disclosed in the aforementioned publications include mercury, mineral oils, halogenated polyethers and polyphenylethers. Owing to their wide availability and their relatively low cost, the mineral oils have become widely accepted as the preferred pump fluid. A typical mineral oil for use as a pump fluid comprises paraffinic, aromatic and naphthenic hydrocarbons.

Most surprisingly, it has now been found that paraffin waxes, when used as pump fluids in diffusion vacuum pumps, result in a significant improvement in the overall performance and efficiency of the pump.

Accordingly, in a first aspect, the present invention provides a process for the generation of a vacuum in a vessel comprising generating a vapour by heating a reservoir of a paraffin wax, discharging the vapour through an inlet into a gas entrainment zone connected to the vessel, thereby generating a vapour jet in the entrainment zone, allowing gas from the vessel to enter the vapour jet, allowing the vapour jet containing the gas to pass to a condensation zone, condensing vapour from the vapour jet in the condensation zone and separately recovering the condensed wax and the uncondensed material.

The paraffin wax used in the process of the present invention preferably comprises greater than 70% wt paraffins, more preferably greater than 80% wt and still more preferably greater than 90% wt paraffins. An especially preferred wax comprises greater than 95% wt paraffins. A most preferred wax is one consisting substantially of paraffinic material. The wax may comprise both straight-chain and branched-chain paraffins, with preference being given for waxes comprising substantial quantities of straight-chain paraffins, in particular greater than 75% wt, more particularly greater than 90% wt straight-chain paraffins. A most preferred wax consists substantially of straight-chain paraffins.

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The paraffins present in the wax may range from C_{15} paraffins to C_{70}^+ paraffins. For use in the process of the present invention, preference is given to waxes comprising paraffins lying within certain molecular weight fractions. A first preferred wax comprises C_{15} to C_{20} paraffins in an amount of at least 60% wt, more preferably at least 75% wt of the total paraffin content. A second preferred wax comprises C_{20} to C_{30} paraffins in an amount of at least 60% wt, more preferably at least 75% wt of the total paraffin content. A third preferred wax comprises C_{30} to C_{40} paraffins in an amount of at least 60% wt, more preferably 75% wt of the total paraffin content. A fourth preferred wax comprises C_{40}^+ paraffins in an amount of at least 60% wt, more preferably at least 75% wt of the tot

The term "paraffin" as used herein is a reference to hydrocarbons or mixtures of hydrocarbons in which the component molecules are substantially aliphatic and are substantially or completely saturated. Preference is given to hydrocarbons or hydrocarbon mixtures in which there is present substantially no ur to a ration.

The wax may have any suitable congealing point, in particular in the range of from 10°C to 120°C, more preferably in the range of from 25°C to 110°C. The wax may have any suitable viscosity for effective operation of the process. In particular, the viscosity of the wax measured at 120°C, may be in the range of from 1 to 20 mm²/s, more preferably from 2 to 15 mm²/s.

It follows from the description of the preferred features of the waxes of use in the process of this invention that many of the waxe will exist as solids or be substantially in the solid phase under conditions of ambient temperature and pressure. However, it will be apparent that, for effective generation of the vacuum, the conditions maintained during the process should be such as to prevent substantial quantities of the wax solidifying.

Waxes of the kind and having the properties as hereinbefore described may be prepared by conventional techniques, for example the conventional refining of crude oil. However, preferred waxes for use in the process of the present invention are synthetic materials, in particular waxes prepared by a Fischer-Tropsch synthesis process.

Fischer-Tropsch synthesis is the name commonly given to processes in which hydrocarbons are prepared from a mixture of carbon monoxide and hydrogen by contacting the mixture at elevated temperature and pressure with a suitable catalyst. Catalysts for use in the Fischer-Tropsch synthesis process frequently comprise, as the catalytically active component, a metal from Group VIII of the Periodic Table of Elemencs. Particular catalytically active metals include ruthenium, iron, cobalt and nickel. Especially preferred waxes for use in the process of the present invention are those prepared by a Fischer-Tropsch synthesis process employing a catalyst comprising cobalt as the catalytically active component.

The catalytically active metal is preferably supported on a porous carrier. The porous carrier may be selected from any of the suitable refractory metal oxides or silicates or combinations thereof known in the art. Particular examples of preferred porous carriers include silica, alumina, titania and mixtures thereof. Silica is a particularly preferred carrier material for the catalyst used in the preparation of the waxes for use in the process of the present invention.

The amount of catalytically active metal on the carrier is preferably in the range of from 3 to 100 pbw per 100 pbw of carrier

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material, more preferably from 10 to 80 pbw, especially from 20 to 60 pbw.

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If desired, the catalyst may also comprise one or more metals or metal oxides as promoters. Suitable metal oxide promoters may be selected from Groups IIA, IIIB, IVB, VB and VIB of the Periodic Table of Elements, or the actinides and lanthanides. In particular, oxides of magnesium, calcium, strontium, barium, scandium, yttrium. lanthanum, cerium, titanium, zirconium, hafnium, thorium, uranium, vanadium and chromium are most suitable promoters. A particularly preferred metal oxide promoter for the catalyst used to prepare the waxes for use in the present invention is zirconium oxide. Suitable metal promoters may be selected from Groups VIIB of VIII of the Periodic Table. Rhenium and Group VIII noble metals are particularly suitable, with platinum and palladium being especially preferred. The amount of promoter present in the catalyst is preferably in the range of from 0.1 to 150 pbw per 100 pbw of carrier.

A particularly suitable catalyst for use in preparing the waxes for use in the process of the present invention is a cobalt/zirconium/silica catalyst. Examples of suitable catalysts which may be used in the preparation of the waxes are disclosed in European Patent Applications publication numbers EP 0 104 672, EP 0 110 449, EP 0 127 220, EP 0 167 215, EP 0 180 269 and EP 0 221 598.

As mentioned, the waxes for use in the process of the present invention may be prepared by the Fischer-Tropsch synthesis, in which a mixture of carbon monoxide and hydrogen is contacted with a catalyst as hereinbefore described. The synthesis is typically conducted at a temperature of from about 125 to about 350°C, preferably from about 175 to 250°C. Typical operating pressures for the synthesis are in the range of from about 5 to 100 bar, more preferably from about 10 to 50 bar. During the synthesis process, the catalyst is typically contacted with a gaseous mixture comprising hydrogen and carbon monoxide in a ratio of less than 2.5, preferably less than 1.75. More preferably, the hydrogen to

carbon monoxide ratio of the mixture is in the range of from 0.4 to 1.5, especially from 0.9 to 1.3.

The Fischer-Tropsch synthesis process, as hereinbefore described, produces waxes suitable for use in the process of the present invention in high yields. The waxes are present substantially in the liquid phase under the conditions prevailing in the process. However, the effluent from the process comprises, in addition to the desired waxes, unconverted feed gas and lighter gaseous and liquid hydrocarbons produced during the synthesis reactions. The waxes may be separated from the unconverted feed gas and the lighter products by conventional separation techniques well known in the art, for example distillation.

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The waxes, once separated from the lighter products may then, if desired, be further refined to yield, for example, one or more of the various wax fractions as described hereinabove. Again, separation techniques for the refining of such waxes are known in the art. Owing to the low the mal stability of the waxes, high temperatures should be avoided during the further refining of the waxes, if cracking of the heavy wax molecules is to be avoided. Accordingly, separation techniques such as vacuum distillation should be applied using, for example the fine vacuum or short-path evaporators known in the art. Typical evaporating equipment for use in such refining processes include the high vacuum falling film evaporators and the high vacuum wiped film evaporators. Typical operating pressures for the aforementioned evaporators range from 10^{-1} to 10^2 Pa.

In a second aspect, the present invention provides the use of a paraffin wax having the properties as hereinbefore described as a pump fluid in a diffusion vacuum pump.

In a third aspect, the prefent invention provides apparatus for the generation of a vacuum comprising a reservoir of a paraffin wax, heating means to generate a vapour from the paraffin wax, a vapour conduit providing a passage for the vapour, a pump vessel, which vessel comprises a gas entrainment zone, a vapour inlet connected to the vapour conduit and of a form whereby vapour

entering the pump vessel is formed into a vapour jet extending into the gas entrainment zone, a gas inlet whereby gas entering the pump vessel is caused to content the vapour jet, a vapour condensation zone arranged to receive the gas and vapour jet leaving the entrainment zone, cooling means to condense vapour in the vapour condensation zone and means to recover separately from the condensation zone the condensed vapour and the gas.

The paraffin wax forming part of the apparatus of this aspect of the invention may be any of the waxes or wax fractions as hereinbefore defined.

The apparatus of the present invention will now be further described, by way of example only, having reference to the attached figure which is a schematic view of an embodiment of the apparatus.

Referring to the figure, a pump vessel 1 comprises a body 2 having the general form of an inverted cone and having a domed upper end 3. A gas inlet 4 is shown in the upper region of the side of the body 2. The vessel (not shown) in which the vacuum is to be generated and maintained is connected to the gas inlet 4 via line 5. A gas outlet 6 is shown in the lower region of the side of the body 2. A vacuum forepump (not shown) is connected to the gas outlet 6 by means of line 7. A vapour inlet 8 is shown located centrally in the upper end 3 of the body 2 and comprises a small aperture through which vapour may pass and enter the upper region of the pump vessel 1. The lower end portion of the body 2 is formed to provide an outlet 9 for condensed wax. The pump vessel further comprises cooling means, represented in the figure as helical tubes 10 extending around the conical outer surface of the body 2. A line 11 extends from the condensed wax outlet 9 to a wax recycle pump 12. A line 13 extends from the wax recycle pump 12 to a wax vaporiser 14. A supply of heating medium to the wax vaporiser 14 is represented in the figure by line 15. A line 16 connects the vapour outlet of the wax vaporiser 14 to the vapour inlet 8 of the pump vessel 1.

In operation of the apparatus represented in the figure, a reservoir of paraffin wax is contained in the lower portion of the

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pump vessel 1 in the region of the condensed wax outlet, line 11, the wax recycle pump 12, line 13 and in the body of the wax vaporiser 14. A second vessel may be provided, for example between the condensed wax outlet 9 of the pump vessel 1 and the wax recycle pump 12, if a greater volume of wax is required in the reservoir.

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The wax in the wax vaporiser 14 is heated by the heating medium to generate a wax vapour. The heating medium may conveniently be hot water or steam contained within the tubes of a conventional shell and tube heat exchanger. Vapour generated in the vaporiser 14 passes along line 16 to the vapour inlet 8 of the pump vessel 1. The vapour passes through a small aperture in the vapour inlet 8, thereby forming a vapour jet 17 in the upper region of the pump vessel 1. The vapour inlet may comprise a plurality of apertures to thereby generate a plurality of vapour jets. In addition, one or more baffles or deflectors may be provided in the entrainment zone to form and guide the vapour jets.

Gas leaves the vessel in which the vacuum is being maintained and enters the upper region of the pump vessel 1 via line 5 and the gas inlet 4. The gas entering the pump vessel 1 contacts the vapour jet 17, diffuses into the jet and is thereby entrained in the jet. The vapour jet 17, together with the entrained gas, travels to the outer region of the pump vessel 1 and contacts the vessel body 2. Under the cooling influence of the cooling tubes 10, the vapour in the region of the vessel body 2 is caused to condense on the inner surface of the body 2. The cooling medium flowing in the cooling tubes 10 is conveniently water. The thus condensed wax flows down the inner surface of the body 2, collects in the lower portion of the pump vessel 1 and eventually leaves the vessel by the condensed wax outlet 9. The condensed wax is recycled via line 11, the wax recycle pump 12 and line 13 back to the wax vaporiser 14.

The gas, together with any uncondensed wax vapour, leaves the pump vessel 1 through the gas outlet 6. Removal of the gas and any uncondensed vapour is effected by the forepump connected to the gas

outlet via line 7. The forepump may be any suitable pump, for example a rotary oil-sealed pump unit.

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The process and apparatus of the present invention as described hereabove may be used to generate and maintain the vacuum in any of the processes known in the art in which operation at reduced pressure is required. Examples include freeze drying, as employed in the food industry, and vacuum distillation applied to the refining of heat sensitive materials, as discussed hereinbefore.

However, it has been found that the process and apparatus of the present invention, relying on paraffin waxes as pump fluids, provide particularly significant improvements in diffusion vacuum pump efficiency and operation, compared with similar pumps relying on conventional pump fluids, when applied to the vacuum distillation of paraffin waxes. This is most surprising, given the existing understanding concerning the operation of the diffusion vacuum pumps. In this respect, reference is made to Ullman's Encyclopaedia of Industrial Chemistry, referred to hereinbefore. Section 21 (Volume B3) of Ullman's Encyclopaedia contains a general description of various aspects of vacuum technology. At subsection 2.3,2.3, of Section 21, a summary of the properties required in a pump fluid for efficient operation of a diffusion vacuum pump is given. In particular, it is specified that the gases being pumped should have a low solubility in the pump fluid. It is, therefore, most surprising that paraffin waxes are excellent pump fluids for use in the pumping of wax vapour.

Accordingly, in a further aspect, the present invention provides a process for the distillation of a paraffin wax comprising generating a vapour by heating a reservoir of a paraffin wax, discharging the vapour through an inlet into a gas entrainment zone, thereby generating a vapour jet in the entrainment zone, evaporating the wax to be distilled in a distillation zone, allowing at least a part of the evaporated wax from the distillation zone to contact the vapour jet in the entrainment zone, allowing the vapour jet containing the evaporated

wax to pass to a condensation zone, condensing vapour from the vapour jet containing the evaporated wax in the condensation zone and separately recovering the condensed wax and the uncondensed material.

The paraffin wax used in the process of this aspect of the invention may be any of the waxes or wax fractions as hereinbefore defined.

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The process for the distillation of a paraffin wax is particularly advantageous when applied in the refining of paraffin wax prepared using the Fischer-Tropsch synthesis process, as hereinbefore described.

In a further aspect, the present invention provides apparatus for the distillation of a paraffin wax, comprising a diffusion vacuum pump, which pump comprises a reservoir of a paraffin wax, heating means to generate a vapour from the paraffin wax, a vapour conduit providing a passage for the vapour, a pump vessel, which vessel comprises a gas entrainment zone, a vapour inlet connected to the vapour conduit and of a form whereby vapour entering the pump vessel is formed into a vapour jet extending into the gas entrainment zone, a gas inlet whereby gas entering the pump vessel is caused to contact the vapour jet, a vapour condensation zone arranged to receive the gas and vapour jet leaving the entrainment zone, cocling means to condense vapour in the vapour condensation zone and means to recover separately from the condensation zone the condensed vapour and the uncondensed material, and an evaporator, which evaporator comprises a distillation zone, heating means for evaporating paraffin wax to be distilled in the distillation zone and an outlet for the evaporated wax, which outlet is connected to the gas inlet of the diffusion vacuum pump.

The paraffin wax forming part of the apparatus of this aspect of the invention may be any of the waxes or wax fractions as hereinbefore defined.

A specific embodiment of the diffusion vacuum pump has been described hereinbefore, having reference to the accompanying figure.

The evaporator may be any of the fine vacuum evaporators or short-path evaporators known in the art and applicable to the distillation of heat sensitive materials, for the example the falling film evaporators or the wiped film evaporators referred to hereinbefore. For the refining of waxes prepared using the Fischer-Tropsch synthesis process, the use of a wiped film evaporator is especially preferred.

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From the description of the apparatus forming the third aspect of the present invention, as schematically represented in the figure, and its operation set out above, it follows that, in the process and apparatus for the distillation of paraffin waxes, the evaporated wax leaving the distillation zone forms the gas referred to hereinabove in connection with the operation of the diffusion vacuum pump. During operation of the process, the evaporated wax diffuses into and is entrained in the vapour jet. This mixture of wax vapours then passes to the condensation zone. It will be appreciated that condensation of both a part of the wax vapour forming the vapour jet and a part of the evaporated wax from the distillation zone may occur.

The operating conditions at any location in the distillation apparatus for the refining of the paraffin wax should be maintained so as to avoid substantial solidification of either the wax being distilled or the wax being used as the pump fluid.

Typical operating conditions of the distillation process for the refining of paraffin waxes are known in the art. The process should be operated at a temperature above that at which the wax begins to solidify. However, the process should not be operated at a temperature at or above the temperature at which the waxes suffer thermal decomposition. Preferred operating temperatures are below 300°C, more preferably below 275°C. The diffusion vacuum pump should preferably be operated to maintain a pressure in the evaporator in the range of from 1 to 20 Pa, more preferably from 5 to 15 Pa.

If various wax fractions are required to be produced, for example the fractions as described hereinbefore, the refining of

the wax feedstock may be effected in a plurality of stages, each stage comprising apparatus and operating under the process regime as described hereinbefore.

In general, it is preferred that, in the processing of waxes, the wax being used as a pump fluid in the vacuum pump is selected to have similar, more preferably substantially the same, properties, for example melting point, carbon number distribution and boiling point range, as the wax vapour being pumped.

Aspects of the present invention will be further described in the following illustrative Example.

EXAMPLE

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A paraffin wax was prepared by means of the Fischer-Tropsch synthesis using the following method:

A cobalt/zirconium/silica catalyst was prepared following the precedure described in European Patent Application publication No. 0 428 223. The catalyst was loaded into a reaction vessel and reduced by contacting the catalyst with a mixture of hydrogen and nitrogen at a temperature of 250 °C, a pressure of 5 bar and a gas hourly space velocity of from 500 to 600 Nl/l/h. The activated catalyst was then contacted with a mixture of carbon monoxide and hydrogen having a hydrogen/carbon monoxide ratio of 1.1 at a gas inlet pressure of from 37 to 39 bar, a temperature of from 210 to 220 °C and a gas hourly space velocity of from 1110 to 1130 Nl/l/h/ The product of the reaction was a mixture of substantially paraffinic hydrocarbons.

The hydrocarbon fraction was subjected to a conventional distillation to remove the C_{20} - components, leaving a C_{21} + hydrocarbon mixture. The resulting mixture was subjected to a vacuum distillation to further refine the C_{21} + hydrocarbon mixture into a range of wax fractions. A light fraction and a heavy fraction, each having a paraffin content of greater than 99%, were selected for further testing. The light wax fraction comprised greater than 90% wt paraffine lying in the C_{18} to C_{27} range. The heavy wax fraction comprised greater than 75% wt paraffins in the range of from C_{28} to C_{40} .

The light wax fraction and the heavy wax fraction were each tested for their performance as pumping fluids in a 18B4A vapour booster pump, commercially available from Edwards High Vacuum International. The pump had the general configuration depicted in the figure. The operating conditions of the pump and performance of the two fractions are summarized in the Table below. In each test, the pump was operated at a power of 6.0 kW. The throughput of the pump was determined for each of the two fractions when generating a vacuum down to each of 0.05 mbar and 0.1 mbar. The pumping speed was determined for each fraction when generating vacuum down to 0.005 mbar.

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For comparison purposes, a similar test was conducted using, as pumping fluid, a commercially available Apiezon oil (AP 201) of the type referred to in US Patent Specification Number 2,560,913, as discussed hereinbefore. The results of this test are also set out in the Table below.

Table

Pumping Fluid	Light Wax Fraction	Heavy Wax Fraction	Apiezon (AP 201)
Throughput (mbarl/s)			
at 0.03 mbar	81	70	50
at 0.1 mbar	92	80	78
Pumping speed (1/s)	9400	9400	4900
at 0.005 mbar			

The claims defining the invention are as follows:

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G-L-A-I-M-S

- 1. A process for the generation of a vacuum in a vessel comprising generating a vapour by heating a reservoir of a paraffin wax, discharging the vapour through an inlet into a gas entrainment zone connected to the vessel, thereby generating a vapour jet in the entrainment zone, allowing gas from the vessel to enter the vapour jet, allowing the vapour jet containing the gas to pass to a condensation zone, condensing vapour from the vapour jet in the condensation zone and separately recovering the condensed wax and the uncondensed material.
- 10 2. A process according to claim 1, characterised in that the wax comprises paraffins in the range of from C_{15} to C_{70}^+ .
 - 3. A process according to either of claims 1 or 2, characterised in that the wax comprises greater than 70% wt, preferably greater than 80% wt, more preferably greater than 90% wt paraffins.
 - 4. A process according to any preceding claim, characterised in that the wax comprises a substantial quantity of straight-chain paraffins, preferably greater than 75% wt, more preferably greater than 90% wt straight-chain paraffins.
 - 5. A process according to any preceding claim, characterised in that the wax is selected from a wax comprising C_{15} to C_{20} paraffins in an amount of at least 60% wt of the total paraffin content, a wax comprising C_{20} to C_{30} paraffins in an amount of at least 60% wt of the total paraffin content, a wax comprising C_{30} to C_{40} paraffins in an amount of at least 60% wt of the total paraffin content and a wax comprising C_{40} + paraffins in an amount of at least 60% wt of the total paraffin content.
 - 6. A process according to any preceding claim, characterised in that the wax has a congealing point of from 10°C to 120°C , preferably from 25°C to 110°C .

- 7. A process according to any preceding claim, characterised in that the wax has a viscosity measured at 120° C of from 1 to $20 \text{ mm}^2/\text{s}$, preferably from 2 to $15 \text{ mm}^2/\text{s}$.
- 8. A process according to any preceding claim, characterised in that the wax is a synthetic wax, preferably one prepared by a Fischer-Tropsch synthesis.
 - 9. A process according to claim 8, characterised in the wax was prepared by a Fischer-Tropsch synthesis using a catalyst comprising cobalt as the catalytically active metal.
- 10. Apparatus for the generation of a vacuum comprising a reservoir of a paraffin wax as defined in any one of claims 1 to 9, heating means to generate a vapour from the paraffin wax, a vapour conduit providing a passage for the vapour, a pump vessel, which vessel comprises a gas entrainment zone, a vapour inlet connected
- to the vapour conduit and of a form whereby vapour entering the pump vessel is formed into a vapour jet extending into the gas entrainment zone, a gas inlet whereby gas entering the pump vessel is caused to contact the vapour jet, a vapour condensation zone arranged to receive the gas and vapour jet leaving the entrainment zone, cooling means to condense vapour in the vapour condensation zone and means to recover separately from the condensation zone the

condensed vapour and the gas.

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11. A process for the distillation of a paraffin wax comprising generating a vapour by heating a reservoir of a paraffin wax as defined in any one of claims 1 to 9, discharging the vapour through an inlet into a gas entrainment zone, thereby generating a vapour jet in the entrainment zone, evaporating the wax to be distilled in a distillation zone, allowing at least a part of the evaporated wax from the distillation zone to contact the vapour jet in the entrainment zone, allowing the vapour jet containing the evaporated wax to pass to a condensation zone, condensing vapour from the vapour jet containing the evaporated wax in the condensation zone and separately recovering the condensed wax and the uncondensed material.

- 12. A process according to claim 11, characterised in that the wax to be distilled has been prepared by a Fischer-Tropsch synthesis.
- 13. A process according to either of claims 11 or 12,
- 5 characterised in that the distillation is effected at a temperature of below 300°C, preferably below 275°C.
 - 14. A process according to any one of claims 11 to 13, characterised in that the evaporator is maintained at a pressure in the range of from 1 to 20 Pa, preferably from 5 to 15 Pa.
- 15. Apparatus for the distillation of a paraffin wax, comprising a diffusion vacuum pump, which pump comprises a reservoir of a paraffin wax as defined in any one of claims 1 to 9, heating means to generate a vapour from the paraffin wax, a vapour conduit providing a passage for the vapour, a pump vessel, which vessel
- comprises a gas entrainment zone, a vapour inlet connected to the vapour conduit and of a form whereby vapour entering the pump vessel is formed into a vapour jet extending into the gas entrainment zone, a gas inlet whereby gas entering the pump vessel is caused to contact the vapour jet, a vapour condensation zone arranged to receive the gas and vapour jet leaving the entrainment zone, cooling means to condense vapour in the vapour condensation zone and means to recover separately from the condensation zone the condensed vapour and the uncondensed material, and an evaporator, which evaporator comprises a distillation zone, heating means for

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- evaporating paraffin wax to be distilled in the distillation zone and an outlet for the evaporated wax, which outlet is connected to the gas inlet of the diffusion vacuum pump.
- 16. Apparatus according to claim 15, characterised in that the evaporator is a falling film evaporator or a wiped film evaporator.
- 17. The use of a paraffin wax as defined in any one of claims 1 to 9 as a pump fluid in a diffusion vacuum pump, in particular in the refining of paraffin wax, especially the refining of a wax prepared by a Fischer-Tropsch synthesis.

18. A process for the generation of a vacuum in a vessel substantially as hereinbefore described with reference to the Example.

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- 19. Apparatus for the generation of a vacuum substantially as hereinbefore described with reference to the accompanying drawings.
- 20. A process for the distillation of a paraffin wax substantially as hereinbefore described with reference to the Example.
 - 21. Apparatus for the distillation of a paraffin wax substantially as hereinbefore described with reference to the accompanying drawings.

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Process And Apparatus For Generating A Vacuum

Abstract

A process for the generation of a vacuum in a vessel comprises generating a vapour by heating a reservoir of a paraffin wax, discharging the vapour through an inlet into a gas entrainment zone connected to the vessel, thereby generating a vapour jet in the entrainment zone, allowing gas from the vessel to enter the vapour jet, allowing the vapour jet containing the gas to pass to a condensation zone, condensing vapour from the vapour jet in the condensation zone and separately recovering the condensed wax and the uncondensed material. Apparatus for the generation of a vacuum comprises a reservoir of a paraffin wax, heating means (15) to generate a vapour from the paraffin wax, a vapour conduit (16) providing a passage for the vapour, a pump vessel (1), which vessel comprises a gas entrainment zone, a vapour inlet (8) connected to the vapour conduit(16) and of a form whereby vapour entering the pump vessel (1) is formed into a vapour jet (17) extending into the gas entrainment zone, a gas inlet (4) whereby gas entering the pump vessel (1) is caused to contact the vapour jet (17), a vapour condensation zone arranged to receive the gas and vapour jet (17) leaving the entrainment zone, cooling means (10) to condense vapour in the vapour condensation zone and means to recover separately from the condensation zone the condensed vapour and the gas. The paraffin wax is preferably a synthetic wax, in particular a wax prepared by a Fischer-Tropsch synthesis.

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