

Oct. 7, 1958

V. MAREŠ

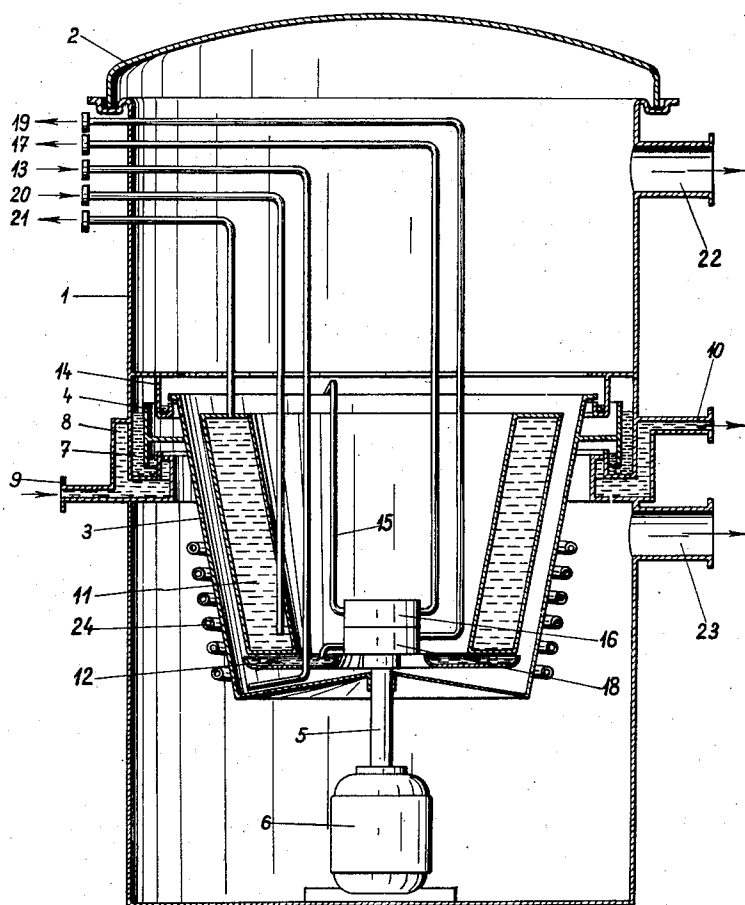
2,855,345

VACUUM DISTILLING APPARATUS

Filed May 11, 1953

2 Sheets-Sheet 1

Fig. 1.



INVENTOR.

Vojtěch Mareš,

BY

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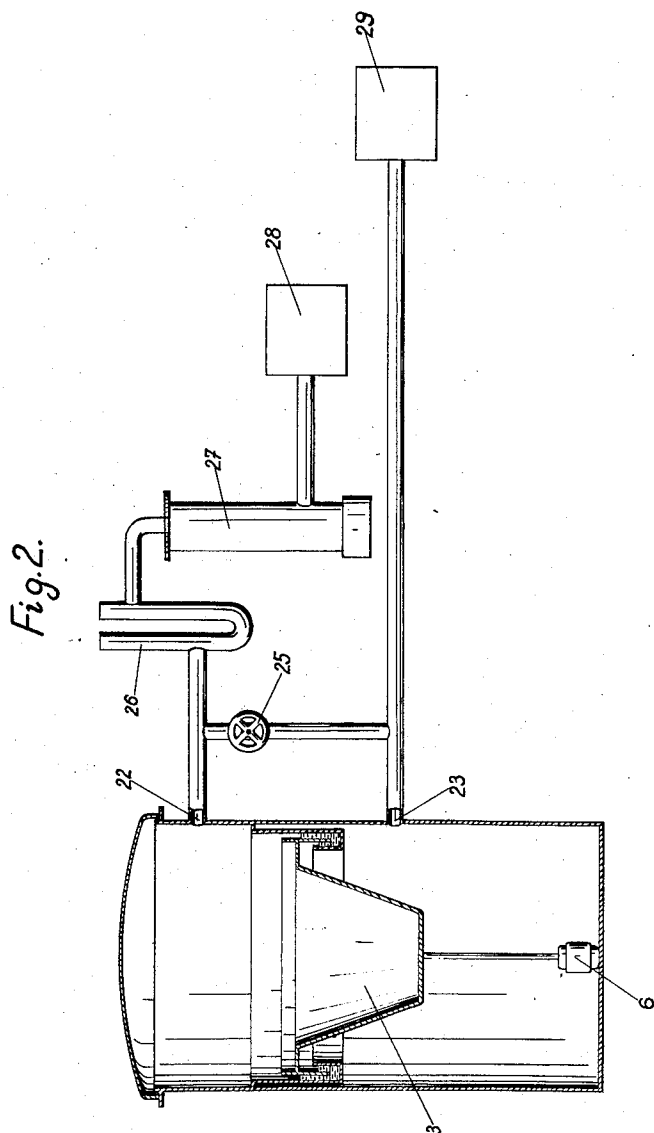
V. MAREŠ

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2 Sheets-Sheet 2



INVENTOR.
Vojtěch Mareš,

BY

Paul H. Simon

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VACUUM DISTILLING APPARATUS

Vojtěch Mareš, Prague-Dejvice, Czechoslovakia, assignor to Zavod Marsala Rybalka, narodni podnik, Decin, Czechoslovakia, a corporation of Czechoslovakia

Application May 11, 1953, Serial No. 354,331

1 Claim. (Cl. 202—205)

High-vacuum distilling apparatuses with free traveling path of the molecules of the evaporated components and running on an industrial scale have been operating satisfactorily only in those cases where the apparatus is of a type provided with a rotary evaporating area on the surface of which the material to be distilled is spread out through centrifugal action. However, even distilling plants of this type are not always convenient in view of the high running expenses owing to the necessary heating and propelling of the rotary vessel placed inside the high-vacuum space. When designing the big industrial units for molecular distillation in the hitherto conventional manner with a centrifugal evaporating area it is moreover necessary to overcome the substantial obstacles met with in solving the problem of a suitable drive arrangement for the rotating evaporating area, said obstacles impeding a satisfactory arrangement of the drive and confining the size of the evaporation area. Therefore it is necessary, in view of the required higher output of the plants for molecular distillation to design the same as a group of distilling apparatuses with lower output. However, such arrangement results in increasing both the operating expenses and investment.

Now it has been disclosed in accordance with the present invention that the above mentioned drawbacks may be eliminated by severing the high-vacuum distillation space from the remaining space of the distillation apparatus by means of a liquid seal constituted by a low-pressure liquid, said liquid seal being maintained under a lower vacuum than the high-vacuum space, the heating contrivance for the rotating evaporation area and the drive of the latter through an electric motor of conventional type being located therein. By such an arrangement it is possible to simplify essentially the construction of high-vacuum distillation apparatuses with a free travelling path of the molecules of the evaporated components and to enable substantial savings in operational expenses to be attained. Furthermore, this novel arrangement enables big units of high-vacuum distilling apparatuses to be erected as there is no substantial difference in the construction of either small or big units if adopting the novel method according to the present invention.

In designing distillation apparatuses with a free travel of the molecules of the evaporated components in accordance with the present invention the usual pressures of 10^{-1} up to 10^{-5} mm. Hg are applied inside the high-vacuum distillation space, mostly a pressure of 10^{-3} mm. Hg while in the remaining vacuum space of the distillation apparatus a pressure is used of the order of several mm. Hg, preferably of 2 to 5 mm. Hg which pressures may be easily obtained and maintained by means of a multi-stage steam ejector or by means of a mechanical oil air pump.

The accompanying drawing illustrates, merely by way of example, an embodiment of the high-vacuum distillation apparatus according to the present invention, with two separate spaces evacuated down to different pressures, Fig. 1 showing diagrammatically the arrangement of the

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apparatus and Fig. 2 its link-up with the respective air pumps.

The apparatus consists of a cylindrical jacket 1 closed on top by a cover and of a vertical shaft 5 propelled by an electric motor 6. The inner surface of the jacket of the rotating vessel 3 forms the evaporation area. The space inside the vessel and above the vessel is being held under a high vacuum and is connected through a branch 22 with a high-vacuum air pump. The high-vacuum space of the distillation apparatus is separated from the remaining vacuum space by a liquid seal 7 filled with an extraneous liquid of a low vapor pressure e. g. apiezone or silicone oil. In order to maintain the lowest possible vapor pressure of the stagnant sealing liquid, the liquid seal is being cooled by a water jacket 8, the cooling water 9 entering at 9 and being discharged at 10. As both spaces of the distillation apparatus are being maintained simultaneously under a vacuum, the sealing liquid column separating the spaces from each other is relatively low since it has to equalize the pressure difference in both spaces attaining merely the value of a few mm. Hg.

The evaporation area proper of the distillation apparatus is constituted by the inner surface of the vessel 3 rotatably mounted inside the high-vacuum space coaxially with the still casing, the distilland or material under distillation being admitted through a pipe 13 and rising through centrifugal effect in the form of a thin skin up to the top rim of the vessel 3. The vessel 3 is heated from outside by heating bodies 24. Inside the vessel 3 an immovable condensing area 11 is provided in the form of a liquid-cooled conical body, the cooling liquid being admitted through a pipe 20 and discharged through another pipe 21. That part of the liquid under distillation which has not been evaporated and passed onto the condensing area 11 mounts up to the top rim of the vessel 3 to be splashed therefrom into an immovable collecting rim or trough 14 and discharged therefrom through the pipe 15 into pipeline 17 by means of a centrifugal pump 16 driven by the extended shaft 5. Likewise the condensate running down the surface of the condensing area 11 is collected in a vessel 12 and discharged therefrom into pipeline 19 through the action of a pump 18 driven by the shaft 5.

The high-vacuum space is connected through a branch 22 with a high-vacuum diffusion air pump, the outer space around the rotating vessel 3 being connected through a branch 23 with an air pump adapted to maintain a reduced pressure of about 2 to 5 mm. Hg, e. g. a multi-stage steam ejector or a mechanically driven oil air pump.

Fig. 2 illustrates schematically the arrangement and connection of the air pumps. The branch pieces 22 and 23 are bridged over by a pipeline and valve 25. The air pump 29 is connected to the pipe attached to the branch piece 23, the pump being adapted to produce and to maintain a reduced pressure of 2 to 5 mm. Hg. The pipe attached to the branch piece 23 is connected with a high-vacuum diffusion air pump 27 provided with an auxiliary air pump 28. Ahead of the diffusion air pump 27 a separator 26 is arranged for catching any gas components taken in and capable of being condensed. Before putting the high-vacuum distillation apparatus into operation both vacuum spaces are pumped out by means of the air pump 29 attached to the low-vacuum space through the branch 23, the valve 25 provided in the by-pass pipe between the low-vacuum and the high-vacuum spaces being open during such operation. On the required operational pressure inside the low-vacuum space being attained, the valve 25 is closed and the auxiliary air pump 28 for the diffusion high-vacuum is put into operation and after an adequately low pressure is attained at the exit of the diffusion high-vacuum pump 27 the diffusion high-vacuum pump 27 itself is put into operation, said pump being connected to

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the branch 22 of the distillation apparatus. Thereupon the electric motor 6 is started for imparting a rotating movement to the evaporation vessel 3 and the distillation vessel is heated.

After having heated the evaporation area up to an adequate temperature, the pre-heated and degasified substance to be distilled is being admitted to the bottom rim of the evaporation area through the pipe 13. Through the effect of the centrifugal force the substance under distillation rises along the evaporation area 3 and is heated during this period up to the distillation temperature and at the same time the volatile components thereof are evaporated and pass unto the condensation surface 11 for liquefying. The condensate flows down the immovable condensation surface 11 and is then discharged into the pipeline 19 through the suction effect of the rotary pump 18 driven by the shaft 5. The remainder of the substance under distillation is being caught up inside the space 14 and discharged therefrom through the pipe 15 and pump 16 into the pipe 17.

When thickening aqueous solutions of thermally unstable substances, a pressure of 5 to 55 mm. Hg is to be used inside the distillation space. In the remaining vacuum-space of the distillation apparatus comprising the heating apparatus for the evaporation area and the driving electric motor either the same pressure is being applied as in the distilling space or a somewhat higher pressure.

The liquid to be evaporated is being admitted through the pipe 13 to the bottom rim of the centrifugal area and through the effect of the centrifugal force is spread in the form of a thin skin along the evaporation area whereupon the thickened liquid passes through the pipe 15 into the rotary pump 16 and is discharged thereby through the pipe 17 out of the distillation apparatus into the adapter. In the course of the rising movement of the substance being thickened along the evaporation area 3 an intense evaporation of the volatile component, e. g. water takes place and the resulting vapors are drawn off through the vacuum pipe connected to the distillation apparatus through the branch 22. A surface or sprinkling condenser of the conventional type is mounted in the vacuum pipe between the air pump and the distillation apparatus.

It is to be understood that instead of the heating elements 24 for heating the evaporation vessel 3 any other convenient heat source may be provided, such as e. g. gas burners.

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In the embodiment illustrated the liquid seal 7 is fitted close to the top rim of the evaporation vessel 3.

It is to be understood that the liquid seal might also be placed at another point of the vessel or on the shaft itself, it being only necessary to locate the whole unit inside the jacket 1 wherein an appropriate reduced pressure is being maintained. Preferably the liquid seal 7 consists of the same liquid which is used for the diffusion air pump 27.

10 What I claim as my invention is:

In a high vacuum still of the type described the improvement comprising a still casing, a vessel rotatably mounted in said casing coaxially therewith and adapted to receive the distilland, means to rotate said vessel, a vaporizing inner surface in said vessel over which distilland is caused to flow by centrifugal force, means for collecting unvaporized distilland from said vaporizing surface; a liquid seal formed by an extraneous liquid having a low vapor pressure and arranged between the inner surface of said casing and said rotatable vessel to divide the space within the casing into an upper chamber containing said vaporizing surface and said means for collecting unvaporized distilland and a lower chamber containing a portion of the outer surface of said vessel and housing the said means to rotate the same; and a first high vacuum pump, a first pipeline connecting said pump with said upper chamber, a second pump for maintaining a lower vacuum, a second pipeline connecting said second pump with said lower chamber, an auxiliary pipe connection between said first and said second pipeline, and closure means in said auxiliary pipe connection to permit to initially evacuate the said upper and lower chambers by the said second pump while the closure means are open and thereafter when the closure means are closed to maintain said second pump a lower vacuum in the said lower chamber and by said first pump a high vacuum in said upper chamber the said lower vacuum being so close to said higher vacuum to prevent the liquid seal from becoming inoperative.

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