March 7, 1944. K. C. D. HICKMAN ET AL 2,343,666

VACUUM DISTILLATION PROCEDURE

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FIG. 1.

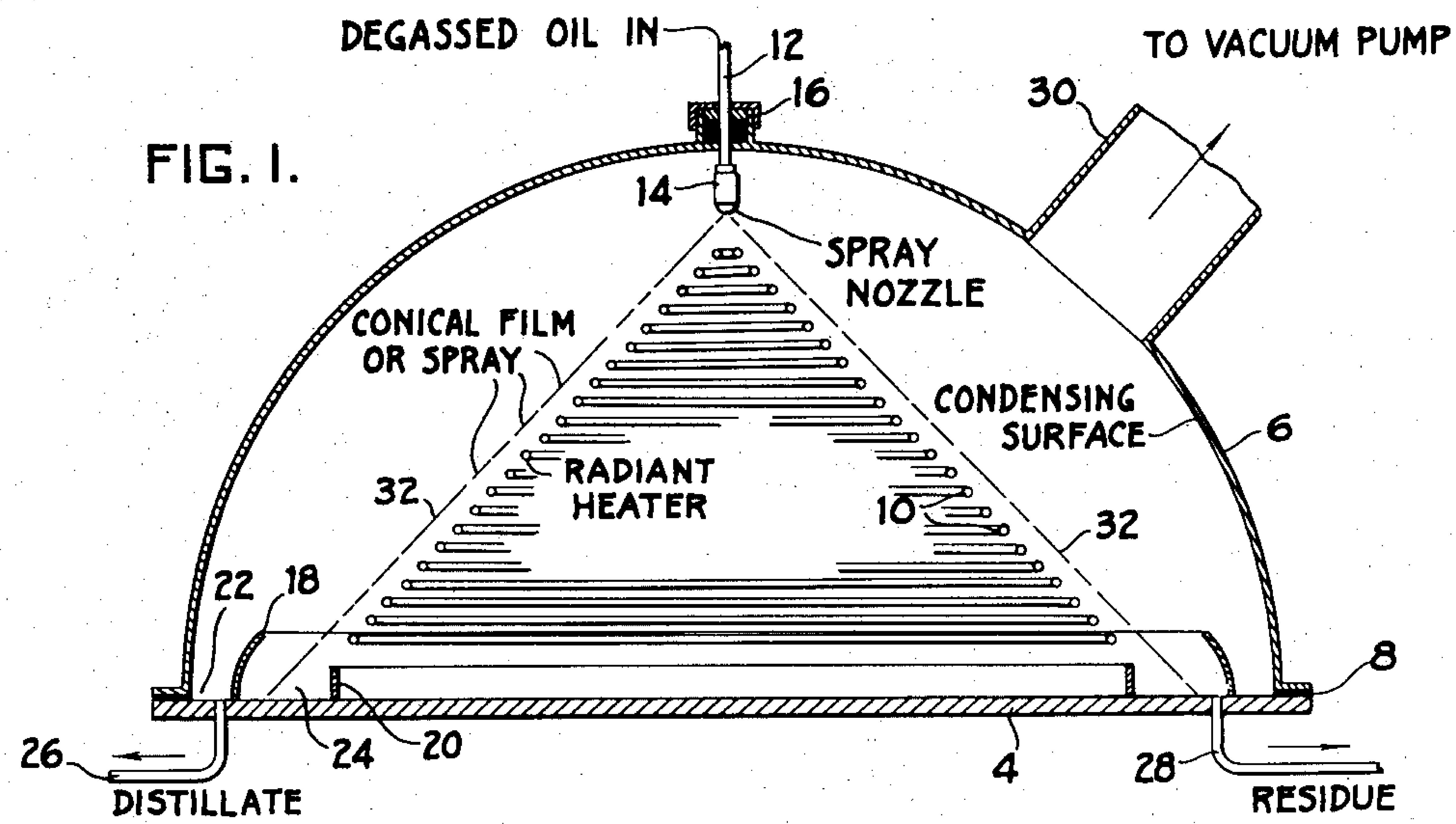


FIG. 4.

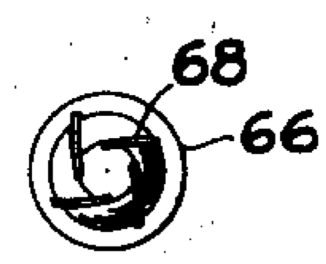


FIG. 3.

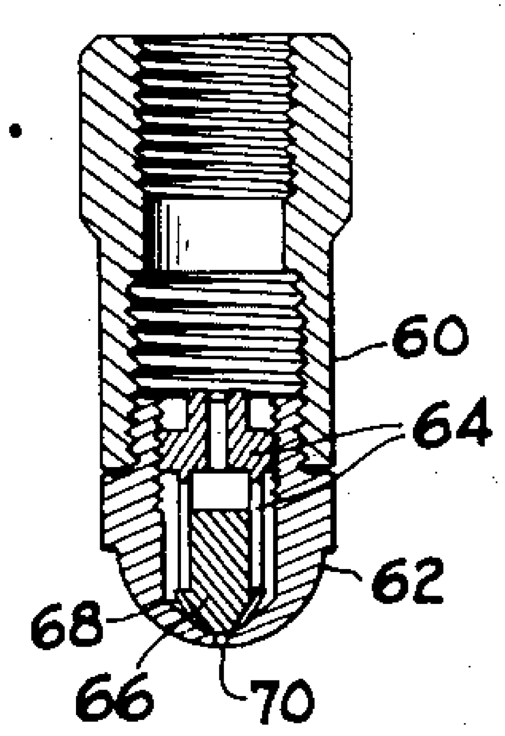
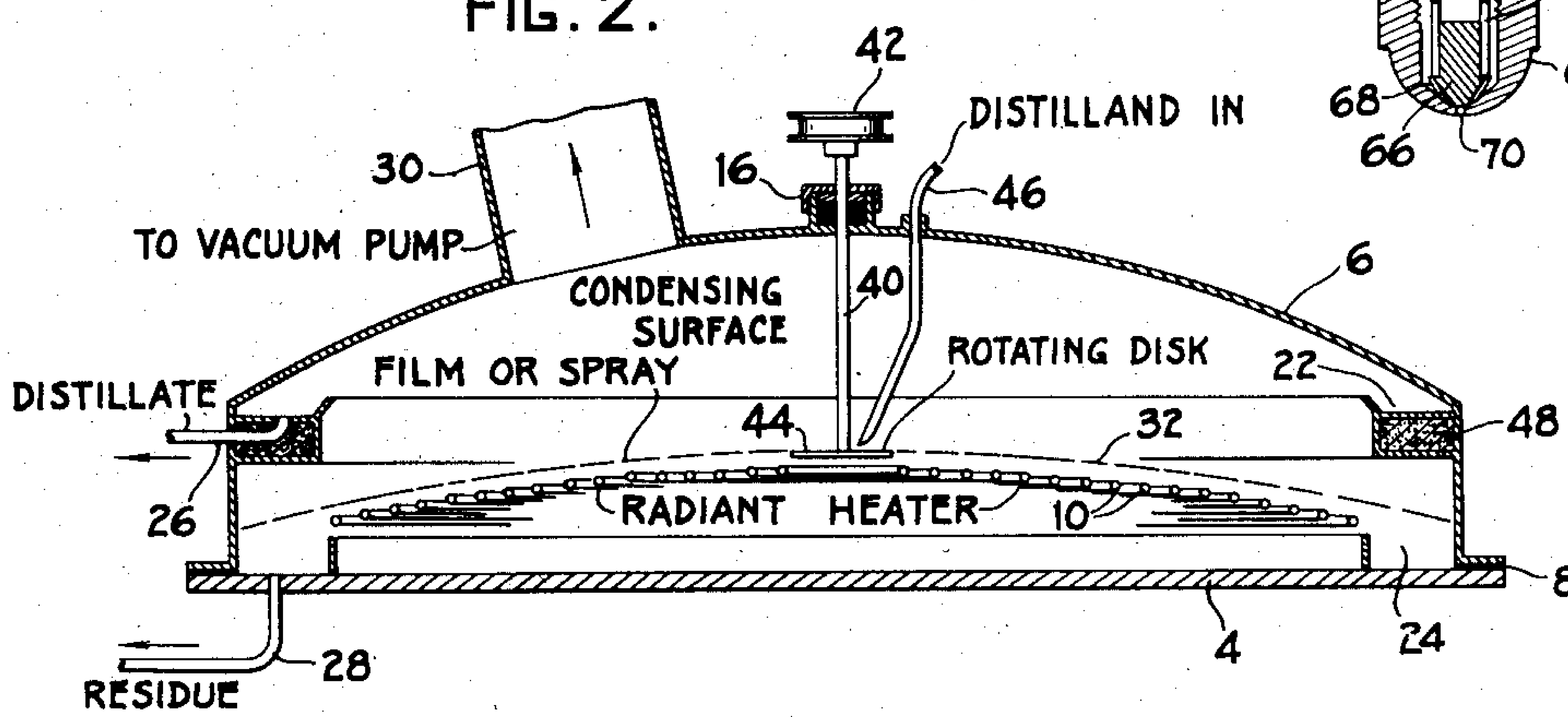


FIG. 2.



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VACUUM DISTILLATION PROCEDURE

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3 Claims. (Cl. 202—52)

This invention relates to improved vacuum distillation process, particularly of the type wherein the substance being distilled and the condensing surface are separated by substantially unobstructed space.

Substances which are ordinarily distilled under vacuum conditions are relatively non-volatile or unstable. In either case, decomposition frequently takes place giving rise to the formation of carbon and other solid matter which deposits upon the vaporizing surface. This requires intermittent shutdowns to clean the still.

This invention has for its object to provide improved vacuum distillation process especially of the high vacuum-unobstructed path type. Other objects are to improve the state of the art. Other objects will become apparent from the following description and claims.

These and other objects are accomplished by our invention which includes adding to the distilland which is to be subjected to vacuum distillation a substance which will increase absorption of radiant energy and then dispersing the distilland into a vacuum distillation chamber where it is heated substantially entirely by radiation to distillation temperature.

In the following description we have given several of the preferred embodiments of our invention, but it is to be understood that these are set forth for the purposes of illustration and not in limitation thereof.

In the accompanying drawing we have illustrated two preferred forms of apparatus in which our invention can be carried out wherein like numbers refer to like parts and wherein;

Fig. 1 is a vertical section of a high vacuum unobstructed path still in which the distilland is formed into a film or film-like spray by spraying it through a spray nozzle, into spaced relation with a radiant heater;

Fig. 2 illustrates a still similar to that of Fig. 1 but shows an alternative method of distributing the distilland in a self-supporting film by centrifugal force;

Fig. 3 is a vertical section of a satisfactory spray nozzle for the apparatus of Fig. 1 and

Fig. 4 is a perspective view of the spray nozzle deflector tip.

Referring to Fig. 1, numeral 4 indicates a relatively flat circular base plate supporting a hemispherically shaped still casing or cover 6, which is maintained in gas tight relation with plate 4 by gasket 8. Numeral 10 designates a radiant heater which may be conveniently made of a spirally wound electrical heating coil. Numeral 12 designates a pipe terminating in spray nozzle 14 which passes through a gas tight packed gland 16. Numeral 18 designates an inwardly curved circular partition integral with base plate 4, and numeral 20 designates a circular partition integral with plate 4 both of which partitions cooperate to form a plurality of gutters 22 and 24. Numeral 26 designates a withdrawal conduit connected to gutter 22 and numeral 28 designates a withdrawal conduit connected to gutter 24. Numeral 30 designates an evacuating conduit which leads to a vacuum pump (not shown). Numeral 32 designates a broken line which is to indicate the path of travel of the spray or film emitted from spray nozzle 14.

Referring to Fig. 2, numeral 40 designates a shaft rotatably mounted in gas tight packing 16, which is provided at the upper end with a driving pulley 42 and at the lower end with a circular disk 44. Numeral 46 designates a conduit passing through the wall of still casing 6 in a gas tight manner, and terminating near the center of plate 44.

During operation of the apparatus illustrated in Fig. 1 liquid to be distilled and containing a radiant heat absorbing material is passed under pressure through conduit 12 and is sprayed through spray nozzle 14 to form a self-supporting film having a conical shape, and indicated by numeral 32. Radiant heater 10 is put into operation in order to heat the film to distillation temperature and the still is evacuated through conduit 30. The cone of distilland 32 is substantially entirely self-supporting and does not materially come into contact with radiant heater 10. However, the heat from radiant heater is absorbed by the film and is thus heated to distillation temperature. Vapors evolved from the film pass to the casing 6 which is air cooled and are condensed thereon. These vapors then flow in the form of liquid condensate into gutter 22 and are removed from the still by way of conduit 26. Undistilled residue is thrown or collects in gutter 24 and is withdrawn through conduit 28.

Spray nozzles are well known which will form a self sustaining film or film of spray and these nozzles form no part of our invention. In Figs. 3 and 4 is illustrated a satisfactory nozzle which is on the market. It comprises a body member 60 into one end of which is screwed the spray nozzle 62. Numeral 64 designates a hollow core in which is slidably mounted a deflector tip 66. Deflector 66 is provided with slits 68 and nozzle

62 with opening 70. Oil under pressure passes through the center of 64, through slits 68 and thence through 70. Slits 68 give a rotary motion to the oil which persists after passage through 70 resulting in a liquid cone. The shape of the cone can be changed by varying the pressure of the oil flowing to the nozzle.

In operating the apparatus illustrated in Fig. 2 the still is evacuated and the radiant heater is put into operation as described in connection with Fig. 1. Distilland which is heat absorbing or which contains heat absorbing materials is introduced through conduit 46. Circular plate 44 is caused to rotate at relatively high speed such as about 200 to 5000 R. P. M. by force applied to pulley 42. The distilland flowing on to the center of plate 44 is caused to flow to the periphery thereof by centrifugal force, and is then flung as a curtain or film to the wall of casing 6. Heat is absorbed by the film from the radiant heater 10 and vapors derived from the heated film condense on the upper part of casing 6, flow into gutter 22, and are removed by way of conduit 26. Undistilled residue is thrown to the lower wall of casing 6 and then flows by gravity into gutter 24 and is withdrawn from the still by way of conduit 28. In order to avoid heating of condensate in gutter 22 by the hot residue which impinges on the lower walls of casing 6, a mass of insulating material 48 is maintained in contact with the base of gutter 22.

In order to increase the amount of radiant energy applied to the film, it is advantageous to utilize polished metal reflecting surfaces. These may be located in the apparatus behind the radiant heaters so that the heat is radiated outward toward the film.

The self sustaining film or layer may be one continuous film of liquid or it may be composed of a body resembling a film in dimensions but composed of separate drops of liquid.

Some distillands naturally absorb infrared wave lengths to a considerable extent, especially if a very thin film is not used. In such cases addition of heat absorbing materials is not necessary. However, such materials may be added to the distilland even if they do exhibit infrared absorption. A very satisfactory heat absorbent which may be added is finely divided or colloidal carbon. Many distillands already contain this material as a decomposition product, in which case it need not be added. Other materials such as powdered or colloidal metals or organic dyes, which absorb infrared, such as colloidal iron, gold, etc. may be used. The absorbing materials should be sufficiently finely divided that they will not interfere with the spray nozzle action or the action of centrifugal force in forming films. Also, they should not be added in amounts sufficient to interfere with this action even when finely divided. The amounts required will vary with the conditions of distillation and the material being distilled. They should be used in amounts sufficient to make the distilland substantially opaque to red and infrared. As a general rule less than one per cent of heat absorbing material will be found to be satisfactory. Larger amounts such as 1 to 10% are useful under special circumstances. Higher or lower amounts can be used and are to be understood as being within the scope of our invention.

Contact of the film with the heater should be avoided as far as possible. This can be done by placing the heater above the spray or by careful adjustment of the spray and suitable spacing of the film from the heater. The same result is accomplished by admitting a small amount of air behind the heaters so that it passes in a direction from the heater to the film and thus prevents contact of the heater and film. The air need only produce a forward stream of a few microns to accomplish this result.

Our invention has the distinct advantage that no vaporizing surface is utilized and, therefore, periodic cleaning thereof is avoided. A particular advantage of the invention is that the distilland is heated to distillation temperature while it is in a form such that rapid vaporization thereof can take place.

What we claim is:

1. The process of high vacuum unobstructed path distillation which comprises passing distilland, in the form of a substantially continuous, physically unsupported film, between, and in spaced relation to, a radiant heater and a condensing surface which heater and surface are separated from each other by substantially unobstructed space, evacuating the space between the radiant heater and the condensing surface, heating the film of distilland to distillation temperature substantially entirely by radiant heat from the radiant heater, condensing the vapors thus formed on the condensing surface and removing condensate and unvaporized distilland from the evacuated space.

2. The process of high vacuum unobstructed path distillation which comprises adding a finely divided heat absorbing agent to the distilland, passing this mixture of absorbing agent and distilland, in the form of a substantially continuous, physically unsupported film, between, and in spaced relation to, a radiant heater and a condensing surface which heater and surface are separated from each other by substantially unobstructed space, evacuating the space between the radiant heater and the condensing surface, heating the film of distilland to distillation temperature substantially entirely by radiant heat from the radiant heater, condensing the vapors thus formed on the condensing surface and removing condensate and unvaporized distilland from the evacuated space.

3. The process of high vacuum unobstructed path distillation which comprises adding a heat-absorbing agent selected from the group consisting of finely divided carbon, finely divided, relatively non-volatile organic dyes and colloidal metals to the distilland passing this mixture of absorbing agent and distilland, in the form of a substantially continuous, physically unsupported film, between, and in spaced relation to, a radiant heater and a condensing surface which heater and surface are separated from each other by substantially unobstructed space, evacuating the space between the radiant heater and the condensing surface, heating the film of distilland to distillation temperature substantially entirely by radiant heat from the radiant heater, condensing the vapors thus formed on the condensing surface and removing condensate and unvaporized distilland from the evacuated space.

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