

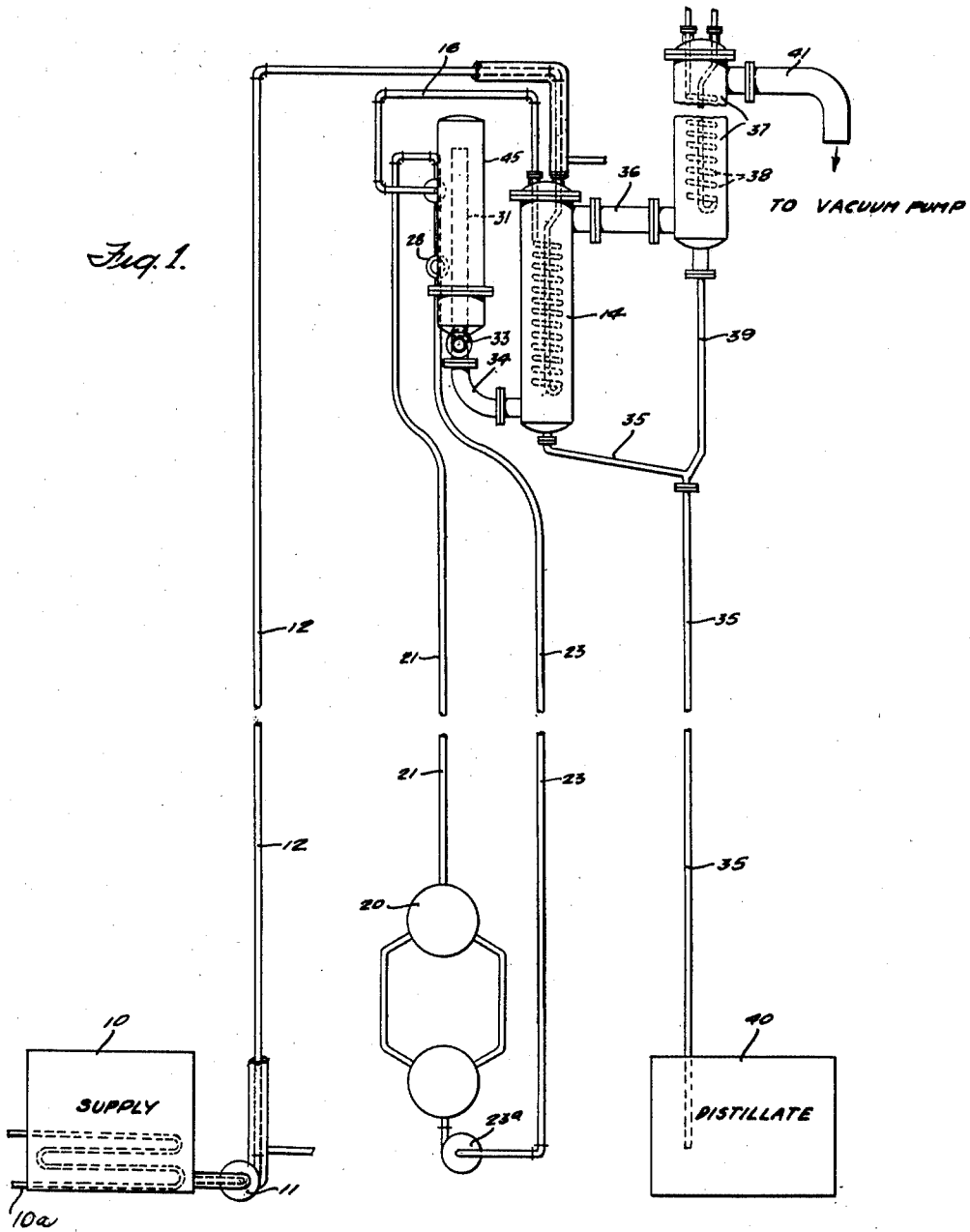
Jan. 10, 1939.

E. M. FRANKEL ET AL
METHOD OF DISTILLATION

2,143,344

Filed April 11, 1935

2 Sheets-Sheet 1



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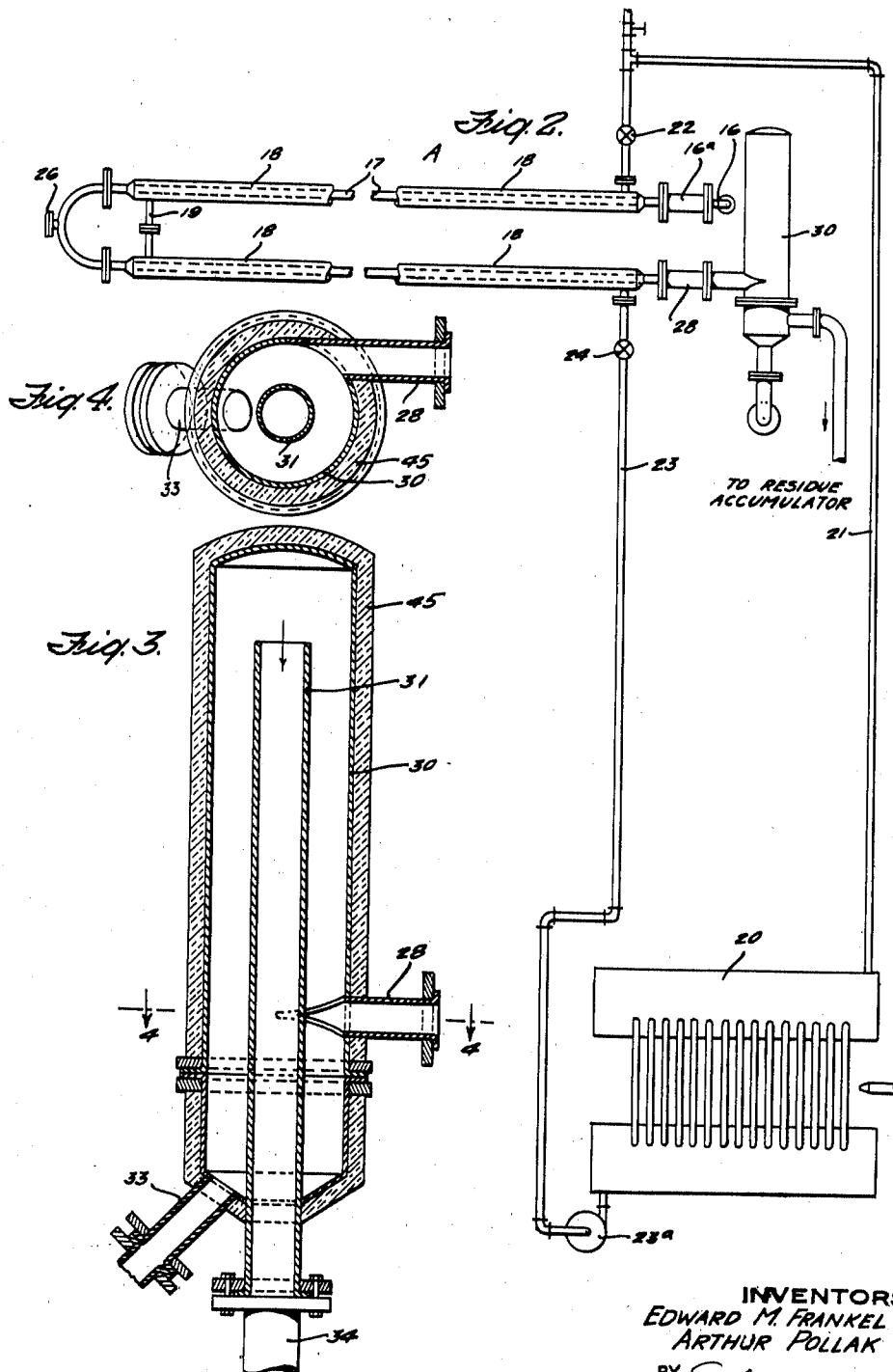
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2,143,344

METHOD OF DISTILLATION

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Application April 11, 1935, Serial No. 15,742

5 Claims. (Cl. 202—52)

Our present invention relates to improvements in methods of distilling high boiling point liquids which are sensitive to temperature at the boiling point. All such materials tend to undergo decomposition at the distillation temperatures. Heretofore it has been usual to use a carrier gas or vapor, as for example a neutral gas or steam, in the distillation for the purpose of raising the total pressure. In our improved process we are able to dispense with the use of such an auxiliary gas or vapor thereby making for high rates of distillation since it will be evident that when an auxiliary carrier is used the volume which it occupies under a high vacuum is relatively very great thereby reducing the capacity of the still.

Our invention is predicated upon the discovery that highly favorable results are obtained in the observance in the distillation of the following conditions: (1) the continuous feeding of a film or fine stream of the liquid through a heating zone, the temperature of which is carefully regulated and in which a suitable vacuum is maintained; (2) the maintenance of a sufficient velocity to prevent overheating and to cause fractional vaporization, the velocity being likewise sufficient to sweep through the zone the unvaporized particles; (3) the physical separation of the liquid and vapor particles as by centrifugal means before opportunity is had for the stream of products to undergo any substantial cooling, followed by the liquefaction of the vapor thus separated in a suitable condenser. Furthermore, not only have we discovered that improved results are obtained without the aid of an assisting gas or vapor but that in most instances greatly improved results are had when the liquid to be distilled is as moisture free as practicable. The manner of carrying out the distillation also precludes the presence of the bulk supply of the liquid in the distillation zone whereby none of it is subjected to the distillation temperature except at the time of distillation. An important corollary of this is that there is substantially no head of liquid at the distillation zone whereby there is no increase of pressure from this source, in contrast to conditions obtaining in distillation in pot stills, for example. To further reduce pressure due to head of liquid, the distilling zones are preferably horizontally disposed. By establishing a barometric leg between the condenser and the accumulator receiving the distillate, the latter may be continuously withdrawn from the system without the aid of pumps.

It is therefore a principal object of the invention to provide for a process of and apparatus

for distilling difficultly distillable liquids as above set forth, many of which have a boiling range as high as 700 degrees F. under comparatively high vacuum. Substances which we have found particularly well adapted for distillation by our improved process and apparatus are the higher fatty acids, glycerides of the fatty acids (fats), hydrocarbons of high molecular weight, the resinous and fatty products derived from wood, especially pine. Among these products last enumerated may be mentioned rosin and the mixture of rosin and fatty acids which are frequently designated tall oil. In its more limited aspects the invention may be said to reside in the improved method of distilling these products.

Other and more specific objects will be apparent as the description proceeds and the features of novelty will be pointed out in the claims.

In carrying out our invention we employ preferably a tube still, the tube or tubes of which is externally heated by the vapor of a substance which has a boiling point at atmospheric pressure approximating somewhat the temperature of the liquid to be distilled. By constituting as the distillation zone a tube which is relatively long in comparison with its diameter and which is surrounded by such a heating vapor or liquid, opportunity is thus afforded for distillation under highly uniform conditions of temperature which may be varied as desired as by the choice of the heating fluid or the pressure thereof and the rate at which it is circulated around the tube. Thus, in the distillation of the higher fatty and rosin acids which takes place mostly between 400 and 600 degrees F. at a pressure below 25 mm. mercury absolute, diphenyl which under atmospheric pressure condenses at 255 degrees C. (491 degrees F.) is recommended. In general, any heating agents may be used which answer these general properties, i. e. which do not undergo appreciable decomposition and may be used under pressures which are not excessive, e. g. greatly in excess of 100 pounds per square inch above atmospheric, further examples of which are diphenyl oxide, naphthalene, various oils, etc. Furthermore, while preferably the heating agent is applied in vapor form so as to take advantage of the heat of condensation, this is not absolutely necessary and the heating agent may be used in the liquid form throughout, if desired.

Our invention will be best understood by reference to the following detailed description taken with the annexed drawings in which

Figure 1 shows in elevation an illustrative embodiment of our improved apparatus;

Figure 2 is a view of the same seen to the left of Figure 1;

Figure 3 is a view on a larger scale and in vertical section of the separator employed; and

Figure 4 is a view taken on section 4-4 of Figure 3.

To illustrate our invention, we may now describe the use of the apparatus for the distillation of the mixture of rosin and fatty acids derived from pine wood in the manufacture of wood pulp by the soda method and commonly designated tall oil, it being understood, however, that the process and apparatus are equally applicable to various other products of a difficultly distillable nature. Such a mixture as recovered from the waste liquor is dark in color, of unpleasant odor and contains impurities of a nature which render it unfit for commercial uses, such impurities also inhibiting crystallization of the rosin acids. Preferably, said oil is rendered substantially anhydrous as by flash distilling the moisture as is more fully described in our co-pending application filed April 11, 1935, Serial No. 15,743.

The crude anhydrous oil, the supply of which is contained in the receptacle 10, is pumped by pump 11 through pipe 12 and through preheater-condenser 14, wherein its temperature is raised, and thence through pipe 16, connection 16a (Figure 2), into the tube still generally denoted A and consisting of an inner tube 17 surrounded by heating jackets 18, 18. Preferably, the tube 17 is horizontally disposed and is of U shape so as to bring the exit of said tube in proximity to the entrance thereof. The heating jacket preferably consists of two parts as shown, one for each leg, such parts being connected by a pipe 19. The tube 17 and jacket 18 may be of any suitable material and for the present purpose we have found copper to be satisfactory. The jacket 18 is heated preferably by means of the vapor of diphenyl or other heating vapor, such vapor being supplied from boiler 20 through pipe 21 having valve 22 into the upper jacket 18 and leaving the lower jacket (as liquid or a mixture of vapor and liquid) through pipe 23 provided with valve 24, through which pipe it is returned by the aid of pump 23a, Figure 1, to boiler 20. The still A is conveniently provided with a thermometer well 26.

In its passage through the tube 17 which is maintained under a suitable vacuum, the crude oil is fractionally vaporized and the mixture of vaporized and unvaporized particles passes through the tube at high velocity through connection 28 into the lower portion of centrifugal separator 30. In the embodiment illustrated, the separator consists of a closed cylindrical vessel having therein an open ended, more or less concentrically disposed cylinder 31. The stream of particles entering through the connection 28 is caused to whirl at high speed in the annular space between the cylinder 31 and the vessel 30, such motion serving to throw the liquid particles against the interior wall of the vessel 30, from which they flow downwardly to the bottom of the vessel and leave the same through exit 33. The whirling stream rises in said annular space, the vapor passing downwardly through the cylinder 31 and leaving the separator through the connection 34 through which it passes to the vapor space of preheater-condenser 14 and in which some of the vapor is condensed, the condensate leaving the condenser through pipe 35 while the uncondensed vapor passes out through pipe 36 to condenser 37 provided with a cooling coil 38. Here the remain-

ing portion of the vapor is condensed and the condensate passes out through pipe 39 joining pipe 35 leading to accumulator 40. The system is maintained under suitable vacuum through the pipe 41 joining the vapor space of the condenser 37, such pipe 41 being connected to a vacuum pump, not shown.

As above mentioned, the condensers 14 and 37 are preferably sufficiently elevated above the accumulator 40 so as to permit the continuous withdrawal of the distillate, the exit pipe 33 for the unvaporized liquid being also connected to a suitable receiving receptacle by means of a barometric leg indicated at 42. (Or, as will be pointed out hereinafter, this residue may be led off to a still of the same type to undergo further distillation.)

The separator 30 and the various pipes for the flow of heated products, including those for the circulation of the diphenyl, are provided with suitable lagging, that of the separator being shown at 45, the other lagging being not shown for the sake of simplicity. Also the tank 10 is preferably supplied with a heating coil 10a while the piping from this tank to the still is preferably steam jacketed, such means permitting the liquefaction of liquefiable solids such as rosin, paraffin, etc., and the feeding of such material in the liquid state to the still.

The tall oil mentioned consists of a mixture of rosin and fatty acids together with some unsaponifiable matter and has a boiling range of 400 degrees F. to 600 degrees F. at below 25 mm. of mercury pressure absolute. For the distillation of tall oil, we have successfully used an average temperature in the heating jackets 18 of from 650 degrees F. to 550 degrees F. while maintaining an absolute pressure on the system of from 25 mm. to 2.5 mm., preference being had for the lower absolute pressure. The calculated velocity in the tube 17 is preferably in excess of 100 feet per second, this value being chosen so as to afford a correct time for heating and to sweep along the unvaporized particles and to provide sufficient velocity in the separator 30 to effect a satisfactory separation of the vapor from the liquid. The upper limit of the velocity will usually be determined by the fact that too great a mass velocity (or momentum) in the separator will cause turbulent flow and hence less separation of the liquid particles. In the case of tall oil distillation, this will be indicated by a darkened distillate.

By virtue of the high velocity through the tubes followed by centrifugal separation, a minimum time is afforded for distillation and a minimum of decomposition results. In the distillation of most organic liquids of high molecular weight and of high boiling point, this time factor is of the utmost importance as otherwise the liquid undergoes decomposition with the formation of gas which thereupon impairs the vacuum causing a rise of the temperature of distillation, thereby increasing the decomposition.

In the case of tall oil, we obtain a light colored, agreeably smelling product which is of sufficient purity so that the solid components consisting chiefly of the rosin acids may upon cooling crystallize, thereby yielding a rosin of exceptional purity.

If desired, one or more stills may be joined together as by leading the liquid particles from the exit 33 to the inlet of an additional still (not shown) of the type described.

Various departures both in the method and apparatus may be made as will occur to those skilled in the art without departing from the spirit of

our invention. As an example, many types of separator may be employed which effect a physical separation of the liquid particles from the vapor by taking advantage of the greater mass and greater inertia of such liquid particles. It is separators broadly of this type that we intend to include by the term "centrifugal" as employed in the claims.

By the term "liquid" in the claims we mean also to include substances such, for example, as rosin which although solid at room temperatures is liquid at or below the distillation temperature.

We claim:

1. The process of distilling difficultly distillable organic substances of the class consisting of fatty and rosin acids to remove therefrom contained impurities, which consists in subjecting a thin continuously flowing film of said substance in a uniformly heated zone under high vacuum to a temperature at which said substance boils, maintaining said substance in said zone until fractions of said substance which it is desired to vaporize attain sufficient vapor pressure to ebulliate, such vaporization taking place under substantially no liquid head and under sufficiently high vacuum to generate a vapor velocity sufficiently great to remove the material from said heated zone without the aid of admixed carrier gases or vapors and before any substantial pyrolytic decomposition thereof results, conducting the stream of the products at high velocity from said zone and thence in a helical path in a separating zone whereby the liquid particles are separated from the vapor by centrifugal action, removing the vapor from the separating zone and condensing same.

2. The process of distilling difficultly distillable higher organic fatty acids which tend to undergo pyrolytic decomposition at their boiling temperature to remove therefrom contained impurities, which consists in subjecting a thin continuously flowing film of the said organic acids in a uniformly heated zone under high vacuum to a temperature at which said organic acids boil, maintaining said organic acids in said zone until fractions of said organic acids which it is desired to vaporize attain sufficient vapor pressure to ebulliate, such vaporization taking place under substantially no liquid head and under sufficiently high vacuum to generate a vapor velocity sufficiently great to remove material from said heated zone without the aid of admixed carrier gases or vapors and before any substantial pyrolytic decomposition thereof results, conducting the stream of products at high velocity from said zone and thence in a helical path in a separating zone whereby the liquid particles are separated from the vapor by centrifugal action, removing the vapor from the separating zone and condensing same.

3. The method of distilling substantially anhydrous tall oil which consists in subjecting a thin continuously flowing film of said tall oil in

a uniformly heated zone under high vacuum to a temperature at which said tall oil boils, maintaining said tall oil in said zone until fractions of said tall oil which it is desired to vaporize attain sufficient vapor pressure to ebulliate, such vaporization taking place under substantially no liquid head and under sufficiently high vacuum to generate a vapor velocity sufficiently great to remove the material from said heated zone without the aid of admixed carrier gases or vapors and before any substantial pyrolytic decomposition thereof results, conducting the stream of products at high velocity from said zone and thence in a helical path in a separating zone whereby the liquid particles are separated from the vapor by centrifugal action, removing the vapor from the separating zone and condensing same.

4. The method of distilling rosin which consists in subjecting a thin continuously flowing film of the said rosin in a uniformly heated zone under high vacuum to a temperature at which said rosin boils, maintaining said rosin in said zone until fractions of said rosin which it is desired to vaporize attain sufficient vapor pressure to ebulliate, such vaporization taking place under substantially no liquid head and under sufficiently high vacuum to generate a vapor velocity sufficiently great to remove said rosin from said heated zone without the aid of admixed carrier gases or vapors and before any substantial pyrolytic decomposition thereof results, conducting the stream of products at high velocity from said zone and thence in a helical path in a separating zone whereby the liquid particles are separated from the vapor by centrifugal action, removing the vapor from the separating zone and condensing same.

5. The method of distilling tall oil which consists in subjecting a thin continuously flowing film of said tall oil in a uniformly heated zone maintained at a substantially uniform temperature in the range of from 650 to 550° F. and under an absolute pressure less than 25 millimeters of mercury, maintaining said oil in said zone until fractions thereof which it is desired to vaporize attain sufficient vapor pressure to ebulliate, such vaporization taking place under substantially no liquid head, whereby under such conditions the vapor particles attain a calculated velocity in excess of 100 feet per second without the aid of admixed carrier gas or vapors, such velocity being sufficient to withdraw the materials from said heated zone before any substantial pyrolytic decomposition thereof results, conducting from said zone the stream of products still under high velocity in a helical path in a separating zone whereby the liquid particles are separated from the vapor by centrifugal action, and then condensing the vapor.

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CERTIFICATE OF CORRECTION.

Patent No. 2,143,344.

January 10, 1939.

EDWARD M. FRANKEL, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, first column, line 25, before the word "oil" insert tall; page 3, first column, line 1, beginning with the words "As an example" strike out all to and including "claims." in line 8; line 12, after "temperature.", insert the following -

When distilling such a solid it is, of course, necessary to liquefy same before admitting it to the still. By "high vacuum" in the claims we refer to absolute pressures in the neighborhood of 25 mm. or under.

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 7th day of March, A.D. 1939.

Henry Van Arsdale.

(Seal)

Acting Commissioner of Patents.