

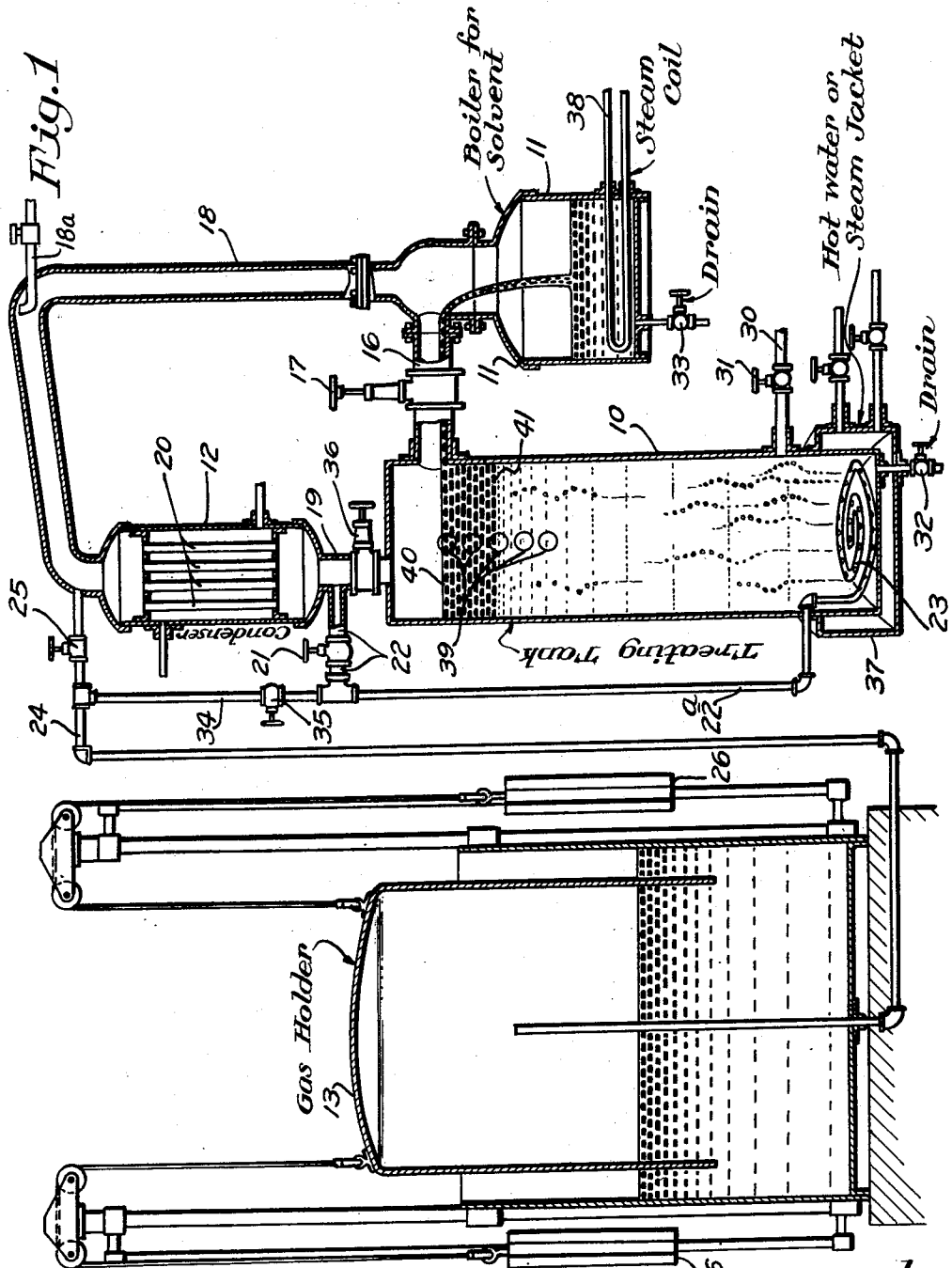
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APPARATUS FOR SAPONIFICATION AND EXTRACTION

Original Filed Jan. 13, 1941 2 Sheets-Sheet 1



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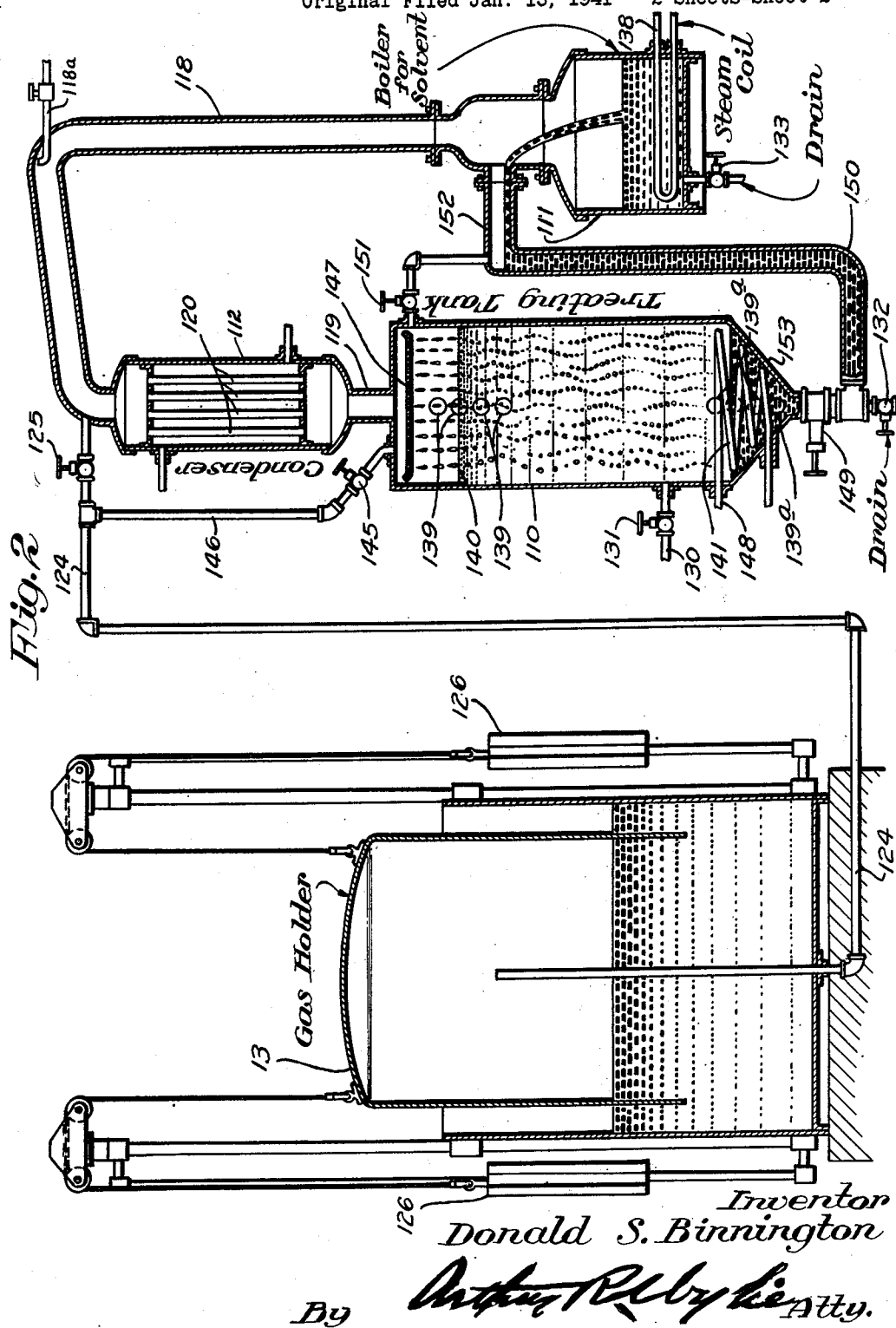
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APPARATUS FOR SAPONIFICATION AND EXTRACTION

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4 Claims. (Cl. 23—267)

This is a division of my application Serial No. 374,261, filed January 13, 1941.

An object of this invention is to provide apparatus for saponifying certain oils, particularly those containing substances subject to oxidation under normal conditions of saponification and extraction, and for extracting non-saponifiable matter under conditions which inhibit oxidation.

Another and more particular object is to provide apparatus for recovering from an oil, particularly a vegetable oil, a non-saponifiable substance containing a vitamin, for example, for recovering from wheat germ oil a non-saponifiable concentrate containing vitamin E, which rapidly loses its potency in the presence of alkali and air, by saponification and extraction of the non-saponifiable substance under conditions which will preserve the potency of the vitamin.

These and other objects as will hereinafter appear are accomplished by this invention which is fully described in the following specification and shown in the accompanying drawings in which the figures show diagrammatically apparatus for practicing the invention. Figure 1 is an embodiment of the invention adapted to the use of a solvent lighter than the liquid to be extracted. Fig. 2 is a modification for the use of a solvent heavier than liquid to be extracted.

In a method of this kind we must consider the use of first a solvent lighter than water and second a solvent heavier than water.

The apparatus illustrated in Fig. 1 for carrying out the first method with a solvent lighter than water comprises principally a treating tank 10, a boiler 11 for solvent, a condenser 12, and a gas holder 13, these being connected by suitable piping which will now be described. The gas holder is maintained under a predetermined slightly super-atmospheric pressure by any well known means. The top of the boiler for solvent is connected with the top of the tank by a relatively large pipe 16 having therein a control valve 17. A vaporline 18 also connects the top of the boiler for solvent through condenser tubes 20 in the condenser 12, a valve 21 and pipe 22 and 22^a to a perforated coil or the like 23 in the bottom of the tank. These perforations enable the solvent to bubble up through the entire body of liquid in the tank. A vent 18^a may be provided to assist in the removal of air from the system prior to the beginning of operations as will appear more fully hereinafter.

The top of the condenser 12 connects through

a pipe 24 with a control valve 25 therein with the gas holder 13, the latter preferably having a suitable counter-weight 26. Liquids may be admitted to the tank through a pipe 30 having a valve 31 therein, and this tank is provided with a drain controlled by a valve 32. The boiler 11 is similarly provided with a drain having a control valve 33 which may also serve for admission of liquids to the boiler. The bottom of the condenser is connected to the top of the tank by a large reflux line 19 controlled by a valve 36. A pressure equalizing line 34 having a valve 35 connects pipe 24 to pipe 22. The connection 22 from line 19 to pipe 22^a should be of large diameter in order to prevent plugging the system with condensed liquid.

The bottom of the tank is provided with a steam or hot water jacket 37 and the boiler 11 is heated as by means of steam coils 38. In this first form, the solvent must be lighter than water in order to effect extraction by separation and may be ethyl ether, petroleum distillate, or similar oil solvents lighter than water and not readily miscible therewith at ordinary temperatures. If some of the solvent does become dissolved in the water in the tank, saturation is soon reached after which the solvent floats on the water with a sharply defined level.

The method of operation of this first method is as follows: The apparatus is completely filled with water and as this is drained off through the drain valves 32 and 33, nitrogen, or a similar inert gas, is admitted to the system from the gas holder 13 at a slightly superatmospheric pressure and until the apparatus is completely filled with this inert gas. When this is accomplished, I pump into the tank 10 the oil to be treated, such as wheat germ oil containing vitamin E, and a suitable alkali solution in proper volume and proportions for saponification. Hot water or steam is introduced to the jacket 37 to heat the mixture of oil and alkali solution, and saponification is accomplished by thus boiling this mixture. During this operation, valves 17, 21, 31, 32, 33 and 35 are closed while valves 25 and 36 remain open so that any vapors distilled off will be condensed and returned to the tank.

After saponification has taken place, the solution in tank 10 is permitted to cool and perhaps is diluted somewhat with water through pipe 30 to raise the level of liquid to the desired point as shown by the sight glasses 39. We are now ready for extraction.

Valves 25 and 36 are now closed and valves 17, 21

and 35 are opened. The boiler 11 is partially filled with a suitable solvent such as ethyl ether or petroleum distillate, and heat is applied to this boiler to cause the solvent therein to distill. The vapor passes through the vapor line 18 to condenser 12, where it condenses and passes down through the pipe 22^a emerging through the perforated coil 23 in small droplets which pass upwards through the soap solution in tank 10 thereby extracting the non-saponifiable constituents. This extract accumulates above the soap solution in the tank until it reaches the level of pipe 16 through which it flows over into the boiler 11. The solvent continues to be volatilized and condensed performing its solvent function until all extractible material has been removed and concentrated in a relatively small amount of the solvent from which it may be recovered by any well-known process.

During this entire time, the space in the upper portions of the apparatus not filled with materials being treated or by the solvent are filled with nitrogen or other inert gas. In this way the material to be recovered, including particularly vitamins, is never in contact with oxygen and consequently may be recovered without loss of potency.

In Fig. 2 is shown a modification of the apparatus for the extraction of non-saponifiable matter by means of a solvent heavier than water. The general arrangement of treating tank 110, condenser 112, boiler 111 and gas holder is much the same as in Fig. 1, but the connections and mode of operation are somewhat different.

A boiler 111 is connected with a condenser 112 by a relatively large vapor tube and the condenser is connected to a tank 110 by a reflux line 119. The treating tank is provided with a conical bottom 153, a coil for steam heating 148, sight glasses 139 and 139^a, a perforated plate 147 for distributing the solvent and a solvent return pipe 150 fitted with valves 149 and 151, and communicating with the boiler 111 through pipe 152. The boiler is fitted with a steam coil 138 and a valve 133 on a drain pipe. The top of condenser 112 is connected to gas holder 13 with a pipe 124 and control valve 125. Communication between the treating tank 110 and the gas holder is established with pipe 146 leading into pipe 124 and fitted with valve 145. Provision for admitting liquids to tank 110 is made with pipe 130 and valve 131, and for drainage through valve 132. Vapor line 118 is likewise provided with a vent 118^a for the same purpose as in Figure 1.

The method of operation is as follows: The entire system is filled with nitrogen or other inert gas by first completely filling with water. This water is drained off through valves 132 and 133; the emptying water being replaced with gas from gas holder 113 through pipe 124. When this operation is completed, oil to be treated, for example wheat germ oil containing vitamin E, is pumped into tank 110 through pipe 130 and followed by alkali solution in proper volume and proportion for saponification. Steam is then introduced through coil 148 to heat the mixture of oil and alkali solution until saponification is completed. During this operation valves 131, 132, 133, 149, 151 and 145 are closed, while valve 125 remains open. Any vapors evolved from the boiling liquid in 110 pass upwards through 119 into condenser tubes 120 where they are condensed to liquid and return to the reaction mixture through 119. Pressure within the system is equalized and maintained slightly in excess of atmospheric by connection to the gas holder.

After saponification is complete the solution in tank 110 is allowed to cool and diluted with water admitted through pipe 130 until the volume is satisfactory as indicated by sight glasses 139. Valve 125 is now closed and valves 145 and 151 opened. A suitable solvent of greater density than water, such as for example carbon tetrachloride or trichlorethylene, is admitted to the boiler 111 through valve 133 and heat is applied through steam coil 138. The vaporized solvent distills through pipe 118, condenses in condensing tubes 120 and flows through pipe 119 to the perforated distributing plate 147. From this plate it falls in droplets through the soap solution dissolving and extracting part of the non-saponifiable matter in its passage and finally accumulating in the lower portion 153 of tank 110. When the interface 141 between the two liquids has reached a suitable point as observed through sight glasses 139^a, valve 149 is opened and the extract permitted to flow back to boiler 111 through pipes 150 and 152. The function of valve 151 is to maintain pressure equilibrium between the boiler and extraction chamber in the event that condenser tubes 120 become overloaded with solvent and completely fill up. Any such variation of pressure between the two parts of the system would result in mechanical displacement of the levels 140 and 141 and might cause the soap solution to be forced over into the boiler.

The extraction process is continued until all the non-saponifiable material has been removed and concentrated in the relatively small amount of solvent contained in the boiler. Recovery of the non-saponifiable from this solution may be effected by known methods.

During the entire time required for introduction of materials, saponification, dilution, cooling and extraction, the vacant spaces of the apparatus are kept filled with nitrogen or other inert gas under a small but constant pressure. In this way the material to be recovered is never exposed to the action of oxygen, particularly while in contact with alkaline soap solutions, a combination which results in rapid destruction of vitamins, particularly vitamin E, with consequent reduction in potency of the concentrate desired.

While I have shown and described but two embodiments of my invention, it is to be understood that it is capable of many modifications. Changes, therefore, may be made which do not depart from the spirit and scope of my invention as disclosed in the appended claims.

I claim as my invention:

1. Apparatus for the saponification of a liquid and for the solvent extraction of the non-saponifiable component thereof which comprises a tank, heat exchange means for said tank, a still disposed separately from said tank, a condenser, a vapor conduit connecting the upper vapor portion of said condenser with the upper vapor portion of said still, a conduit for condensate connecting the lower portion of said condenser with the upper portion of said tank, and a valved conduit connection between the tank and the still, below said vapor conduit connection so arranged as to permit gravity return flow from said tank to said still.

2. Apparatus for the saponification of a liquid and for the solvent extraction of the non-saponifiable component thereof which comprises a tank, heat exchange means for said tank, a still disposed separately from said tank, a condenser, a vapor conduit connecting the upper vapor portion of said condenser with the upper vapor por-

tion of said still, a conduit for condensate connecting the lower portion of said condenser with the upper portion of said tank, and a valved conduit connection from the bottom of said tank to said still so arranged to permit gravity return flow from said tank to said still.

3. Apparatus for the saponification of a liquid and for the solvent extraction of the non-saponifiable component thereof which comprises a tank, heat exchange means for said tank, a still disposed separately from said tank, a condenser, a vapor conduit connecting the upper vapor portion of said condenser with the upper vapor portion of said still, a conduit for condensate connecting the lower portion of said condenser with the upper portion of said tank, conduit means extending from the bottom of said condenser to the bottom of said tank, and valved conduit

means near the top of said tank connecting said tank and said still.

4. Apparatus for the saponification of a liquid and for the solvent extraction of the non-saponifiable component thereof which comprises a tank, heat exchange means for said tank, a still disposed separately from said tank, a condenser, a vapor conduit connecting the upper vapor portion of said condenser with the upper vapor portion of said still, a source of inert gas, a conduit connecting said source and said condenser, a conduit for condensate connecting the lower portion of said condenser with the upper portion of said tank, and a valved conduit connection between the tank and the still, below said vapor conduit connection so arranged as to permit gravity return flow from said tank to said still.

DONALD S. BINNINGTON.