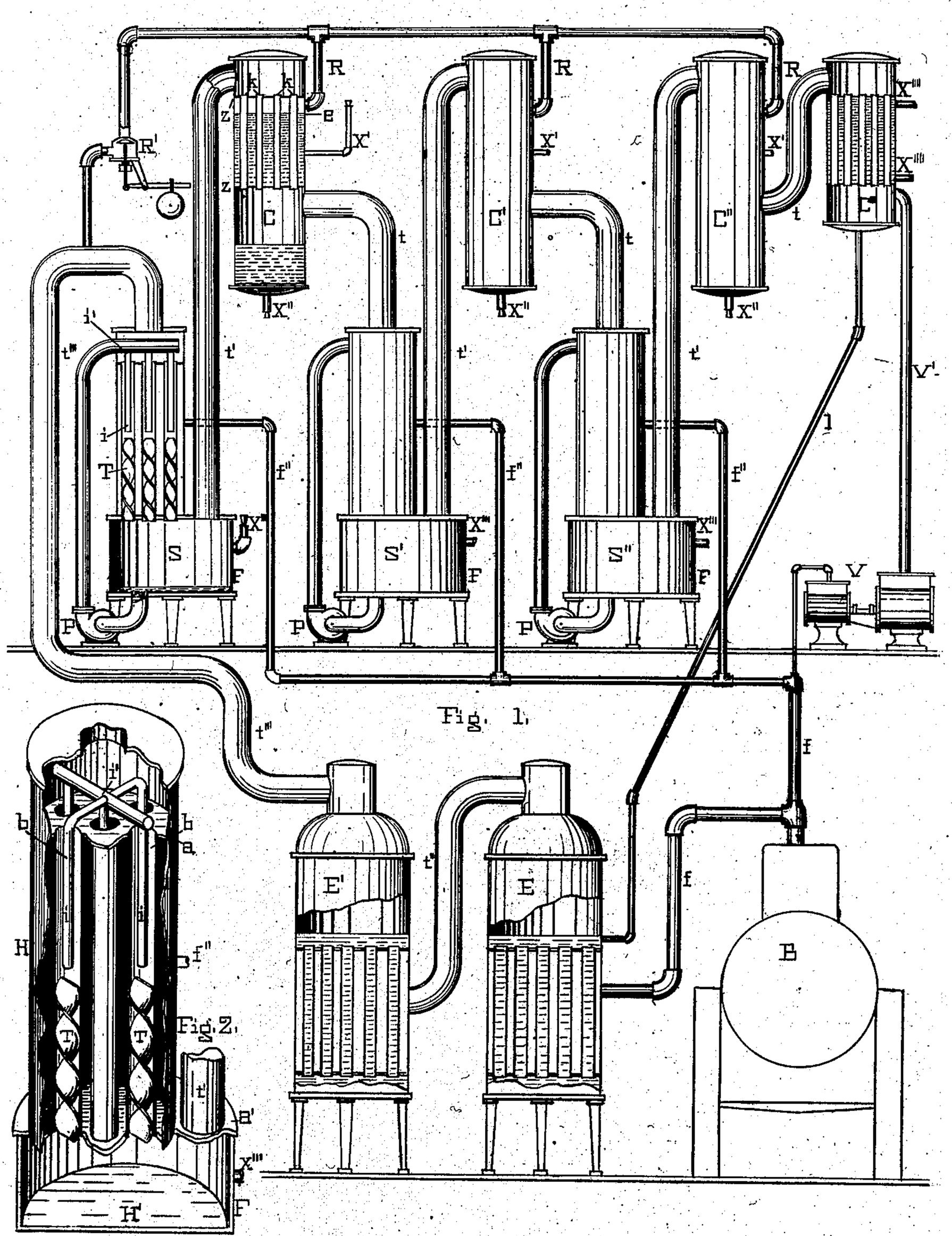
No. 881,525. PATENTED MAR. 10, 1908.

F. J. WOOD.

PROCESS OF DISTILLING GLYCERIN.

APPLICATION FILED MAR. 22, 1905.



Witnesses:
Andrew O. Jackson
George MacDonald

Inventor.
Frank J. Wood

UNITED STATES PATENT OFFICE.

FRANK J. WOOD, OF NEW YORK, N. Y., ASSIGNOR TO MARX AND RAWOLLE, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

PROCESS OF DISTILLING GLYCERIN.

No. 881,525. Specification of Letters Patent. Patented March 10, 1908.

Application filed March 22, 1905. Serial No. 251,520.

To all whom it may concern:

Be it known that I, FRANK J. WOOD, a citizen of the United States, residing in the city of New York, State of New York, have invented a certain new and useful Improved Process of Distilling Glycerin, of which the following is a specification.

In distilling glycerin according to the processes followed heretofore it has been customary to employ a single still, or a plurality of stills each having a separate steam supply.

So far as I am aware there has been no process developed which contemplated the employment of a plurality of stills connected in series and successively supplied with a single column of steam to be mingled with the glycerin contained in the several stills. The reason for this is that it has been customary to introduce or inject the superheated steam into the glycerin through a pipe entering the still at the bottom thereof or beneath the surface of the body of glycerin. Consequently the line of communication through a series of such stills would also be through the bodies of glycerin in the stills, and therefore a single column of steam to be intimately mingled with the glycerin in all of the stills in succession, would have to be under high pressure in order to overcome the hydrostatic pressures of the bodies of glycerin. This would obviously cause the glycerin to be vaporized very slowly unless it were heated to a degree which might occasion its decomposition. These facts alone would preclude supplying a single column of steam to a series of stills in which the steam and glycerin were mingled in the manner stated. But aside from these points, although of equal importance, is the fact that where a plurality of stills is employed a uniform degree of vacuum is essential. This should be about 28 or 29 inches since that pressure will permit of effectively distilling glycerin while avoiding its decomposition. The action of a vacuum pump connected to a series of stills of the known construction would be against the bodies of glycerin contained in the stills. Consequently while the desired vacuum, or even a perfect vacuum, might be created in the last still, or that adjacent the pump, there would be material differences in the degrees of vacuum in the preceding stills, varying according to the heights of the several bodies of glycerin, with the probable result that in the first still the extent to which the pressure would be relieved would not be sufficient to avoid decomposition.

The essence of my invention is economy of steam supply and consequent saving of fuel necessary to distil a given quantity of glycerin. In carrying out the process I am enabled to employ a plurality of stills and condensers connected in series and to which a single column of steam is supplied, by so mingling the steam with the several bodies of glycerin that its passage into and through the stills is accelerated rather than retarded. The economy in steam supply is aided by utilizing the heat given off by the glycerin in its condensation to generate steam which may be added to that supplied to the stills.

In the accompanying drawing I have shown appropriate mechanism for carrying out the process, Figure 1 illustrating the entire system in conventional arrangement, and Fig. 2 a detail of one of the stills.

Referring to the drawing, E and E′ are evaporators in "double effect" arrangement.

S, S′, S″ are stills, each comprising a glycerin reservoir F supplied at X‴ and on which is mounted a cylindrical casing H containing vertically disposed tubes *b*, supported by plates *a*, *a*′. The tubes of the first still communicate through an opening in the top of the casing with a pipe *t*‴ through which the steam is supplied from evaporator E′. In the lower portions of the tubes are spiral plates T while above the plates are the extremities of nozzles *i* extending downwardly in the tubes *b* from a manifold *i*′, in communication with the reservior F, a centrifugal pump P supplying glycerin to the manifold and nozzles. The normal level of the glycerin in tanks F is well below the tops of the latter so that ample space is left for the passage of the steam and glycerin vapors from the tubes *b* to the pipes *t*′ leading to the glycerin condensers C, C′, C″. Through pipes *f*″ high pressure steam from boiler B enters the interior of the casings H and surrounds the tubes *b*.

The glycerin condensers C, C′, C″ contain plates Z inclosing a space for hot water supplied through pipes X′. Tubes *k* are vertically mounted in the plates Z and are thus surrounded by the hot water and afford communication from pipe *t*′ to the space in the condensers beneath the lower plates Z constituting distillate tanks having outlets X″. Pipes *t* connect the distillate tanks

with the succeeding stills S′, S″. From points above the water level *e* of the hot water spaces of these condensers pipes R are shown leading to the steam pipe *t‴*, a pressure regulator R′ being indicated near the intersection. The steam and glycerin condenser C‴ is of substantially the same construction as the glycerin condensers except that it is supplied with a circulation of cold water by pipes X⁗. A pipe 1 establishes communication between this condenser and evaporator E, and a pipe V′ connects it with the vacuum pump V.

Preparatory to beginning the process the tanks F of the stills are supplied to about two-thirds of their capacity with hot glycerin. The vacuum pump is put in operation to create vacuum of about 28 inches in the stills their tanks and tubes, and in the tubes of the condensers and in the evaporator E′. This uniform vacuum may readily be established throughout any number of stills and condensers since the line of communication from the evaporator E′ to the pump is not at any point obstructed by a body of glycerin. By adjustment of the pressure regulator R′ the vacuum in the hot water spaces of the condensers C, C′, C″, is reduced to about 16 inches so that the water will boil at about 175 degrees F. I have selected this degree of pressure for the reason that if the pressure in the hot water space of the condensers C and in pipe R were to be the same as that in the stills, 28 inches, the water would boil at a very low temperature or about 101 F. This would create a lower temperature in tubes *k* than is necessary for condensing the glycerin, and as the steam and vapors passed through the tubes the steam would be cooled to such an extent that a great quantity of heat would be required to superheat it before it would be available for distilling in the succeeding still, since for that purpose its temperature should be between 310 and 350 degrees F. If, on the other hand, the pressure in the vapor spaces of the condensers was too great the steam and glycerin passing through the tubes would be at such a high temperature that all of the glycerin might not be condensed. Consequently I have determined that a temperature of about 175 degrees F. is proper since at that temperature more heat will be maintained in the steam and yet the glycerin will be fully condensed. To obtain this temperature a pressure of about 7 pounds absolute or vacuum of about 16 inches in pipe R is essential. I accordingly weight the pressure regulator R′ so that with 28 inches of vacuum, or about one pound absolute, in the stills, it will be seated as against a pressure of 7 pounds or less in pipe R. When the vacuum pump is started it exhausts the air from the stills, their tanks and tubes, the tubes *k* of the condensers and the evaporator E′, but the pressure in pipe R will not be affected by reason of the weight which holds the regulator valve to its seat. But when the pressure in the stills has been reduced to about 6.7 pounds (the approximate difference in the pressures) the pressure in pipe R will unseat the valve and this pressure will be lessened until it is approximately 6.7 pounds higher than that of the stills. As the lessening of the pressure in the stills continues the pressure in pipe R also continues to be lessened, but the difference of approximately 6.7 pounds between the two pressures remains constant. Consequently when the desired vacuum of 28 inches in the stills is obtained the vacuum in pipe R will be about 16 inches.

In commencing the process it will be assumed that the evaporator E contains "sweet water" from a previous distillation supplied to it through pipe 1 from the steam condenser C‴. It is desirable that the impurities contained in this sweet water shall not become mingled with the glycerin to be distilled but that only steam generated from pure water be used. I therefore supply the evaporator E′ with pure water. Steam from the boiler B vaporizes the sweet water in evaporator E and this vapor passes to the steam space of evaporator E′ and vaporizes the pure water therein, the pure steam generated by this double effect evaporating system being conducted through pipe *t‴* to the first of the stills and there mingled with the glycerin.

It may here be noted that although the steam supplied to the evaporator E from the boiler is at the same temperature, and is, in fact, the same steam, as that employed for distilling the glycerin in the stills, yet the glycerin contained in the sweet water will not be distilled in the evaporator E for the reason that it is combined with water which will evaporate first at temperatures from 110 to 160 degrees F., while the glycerin requires a vaporizing temperature of 280 degrees F. or more, and for the further reason that the evaporator E is being continually supplied with the sweet water. When the evaporator E has operated sufficiently long the supply of sweet water and of steam may be cut off and the accumulated glycerin withdrawn.

The pure steam from the evaporator E′ passes through pipe *t‴* to the still S and entering at the top of the casing H passes down the tubes *b*. The pump P is started and the glycerin is thereby withdrawn from tank F and enters the manifold and nozzles *i*. During its gravitation the glycerin is heated, and the steam jet is superheated by steam under about 150 pounds pressure passing from the boiler through pipe *f‴* into the space of the cylinder H around the tubes *b*, which are thus heated to about 370 degrees F. On passing out of the nozzles the glycerin unites

with the steam and their combined vapors under the action of the superheating steam pass over or around the spiral plates T where they are subjected to a whirling action which effects their complete physical association and vaporization of the glycerin. The steam and glycerin vapors pass out of the tubes *b* into the tank F and then through pipe *t'* to the condenser C, the unvaporized glycerin and residue dropping back into the tank F. The temperature of the water contained in condenser C is about 175 degrees F. which, under the vacuum there maintained, is sufficiently cool to condense the glycerin vapors but sufficiently hot to maintain the steam vapors, as previously explained. Consequently the distilled and condensed glycerin falls to the receptacle at the bottom of the condenser from which it may be withdrawn through pipe X'', while the steam passes through pipe *t* to the still S' in which are repeated the described steps of withdrawing glycerin from the reservoir, its mingling with the steam, and superheating of the steam.

While I specify superheating the steam at each distillation, yet the essential office of the high pressure steam supplied through pipes *f''* is the heating of the glycerin, as the steam would be available for continued and successive use so long as it remained above the saturation point, and would not itself require superheating before mingling with the glycerin at each distillation. From the foregoing it will be seen that any number of stills might be employed in each of which a body of glycerin could be distilled with only one column of steam for successively mingling with the glycerin in the several stills, as the steam would be of the same value in all of the stills. By my invention, however, I may do more than maintain the steam supply brought to the first still from the evaporator. I may increase its volume by adding to it steam generated in the hot water spaces of the condensers C, C', C''. In vaporizing the glycerin absorbs the heat of the high pressure steam supplied through pipes *f''* and consequently it will give up this heat when it condenses. As it condenses by its passage through the tubes *k*, which are surrounded by the water at a temperature to effect its condensation, this water will take up the heat given off by the glycerin and will attain the boiling point, the steam generated passing through pipe R to pipe *t'''* for use in the stills. This auxiliary steam supply, may, however, be confined within pipe R until it has attained a certain degree of pressure for which the regulator R' may be adjusted, since its confinement tends to maintain the temperature of the water in the condenser. Obviously the steam thus generated in the condenser may be conducted elsewhere than to the steam-passing to the stills.

The actions in the sills S', S'', and their respective condensers are the same as those stated with reference to still S and its condenser, each still containing glycerin which is vaporized and mingled with a single column of steam, the flow of the steam being in the same direction as the gravitating columns of glycerin with which it unites, its value being maintained throughout the system, only its temperature varying since each glycerin condensation lowers it from the superheated to near the saturation point, and each condenser collecting distilled and condensed glycerin while maintaining the steam and generating additional steam by the condensation of the glycerin.

From the last of the glycerin condensers C'' the steam passes to the cold water condenser C''' where the steam and the glycerin which may be entrained with it are condensed, after which the sweet water passes to the evaporator E as before described.

I am aware that there have been various means devised and practiced for separating or extracting glycerin from solids or liquids in which it is contained, such as fats, the residue from soap manufacture, vinasses, etc., and that in some of the modes of treating these residues steam has been injected into or mingled with the glycerin-containing materials other than against the resistance of the hydrostatic pressure of the material, but these known methods merely separate glycerin which must be subsequently distilled to derive what is known as chemically pure glycerin, and are therefore not germane to the multiple effect of the steam supply, that is, the utilization of a single jet of steam for successively combining with, and effecting the distillation of, the glycerin contained in a plurality of stills.

I claim as my invention:—

1. The process of effecting a series of glycerin distillations, consisting of successively combining a plurality of bodies of glycerin with a single jet of steam flowing through all of the stills in which the bodies of glycerin are contained, and condensing the glycerin vapors obtained from each body of glycerin.

2. In the process of distilling glycerin, successively combining a single column of steam with a plurality of bodies of glycerin, and condensing the vaporized glycerin of each combination while maintaining the steam, the latter combining with a succeeding body of glycerin.

3. The process of distilling glycerin consisting of utilizing a single jet of steam for a series of successive distillations, combining with the steam jet in each distillation a flowing column of glycerin, and condensing the glycerin vapors of each distillation while maintaining the steam, the latter combining with a succeeding column of glycerin.

4. The process of distilling glycerin consisting of heating columns of glycerin in se-

ries, successively combining with the columns of glycerin a single jet of steam, and condensing the vaporized glycerin of each column while maintaining the steam, the latter combining with a succeeding column of heated glycerin.

5. The process of effecting a series of glycerin distillations consisting of successively combining flowing columns of glycerin with a single jet of steam, superheating the combined vapors at each distillation, and condensing the glycerin vapors obtained from each column of glycerin.

6. A step in the process of distilling glycerin, consisting of generating steam by the heat given off by the glycerin vapors in the condensation thereof.

7. In a glycerin distilling process, vaporizing the glycerin, condensing the vapors, generating steam by the heat given off by the glycerin in its condensation, and utilizing in the distillation system the steam thus generated.

8. The process of distilling glycerin, consisting of combining steam with a column of glycerin flowing by gravity, superheating the steam, condensing the vaporized glycerin, generating steam by the condensation of the glycerin vapors, and utilizing in the distillation system the steam thus generated.

9. The process of distilling glycerin, consisting of heating a flowing column of glycerin, combining steam with the glycerin, superheating the steam, condensing the glycerin in a hot water condenser, and conducting the steam generated by the condensing glycerin to the steam prior to its union with the glycerin.

10. The process of distilling glycerin consisting of utilizing a single jet of steam for a series of successive distillations, combining with the steam jet in each distillation a column of glycerin flowing in the same direction as the steam jet, superheating the steam at each distillation, condensing the glycerin vapors of each distillation, and adding to the steam prior to its union with the first column of glycerin steam generated by the glycerin in each condensation thereof.

11. The process of distilling glycerin consisting of utilizing a single jet of steam for a series of successive distillations, combining with the steam jet in each distillation a column of glycerin flowing in the same direction as the steam jet, superheating the steam at each distillation, condensing in a hot water condenser the vapor obtained from each column of glycerin, and conducting the steam generated by the condensing glycerin to the steam prior to its union with the first column of glycerin.

12. In the process of distilling glycerin, the following steps: condensing the steam and glycerin from a previous distillation, evaporating the sweet water thus obtained, and generating pure steam by the heat of the vapors of the sweet water.

13. In the process of distilling glycerin, the following steps: condensing the steam and glycerin from a previous distillation, evaporating the sweet water thus obtained, generating pure steam by the heat of the vapors of the sweet water, and conducting the pure steam to the still.

14. The process of distilling glycerin consisting of combining steam with glycerin and superheating the combination in the presence of a vacuum, and condensing the glycerin out of the steam by causing its heat to be absorbed by a body of water which is in the presence of a vacuum less than that under which the glycerin is distilled, and utilizing in a succeeding distillation the steam maintained during the condensation of the glycerin.

15. The process of distilling glycerin consisting of combining steam with glycerin and superheating the combination in the presence of a vacuum of approximately 28 inches of mercury, condensing the glycerin by causing its heat to be absorbed by a body of water which is in the presence of a vacuum of approximately 16 inches of mercury whereby steam is generated from the condenser water, and utilizing the steam thus generated in the distillation of the glycerin.

FRANK J. WOOD.

Witnesses:
ANDREW C. JACKSON,
GEORGE MACINTOSH.