

(No Model.)

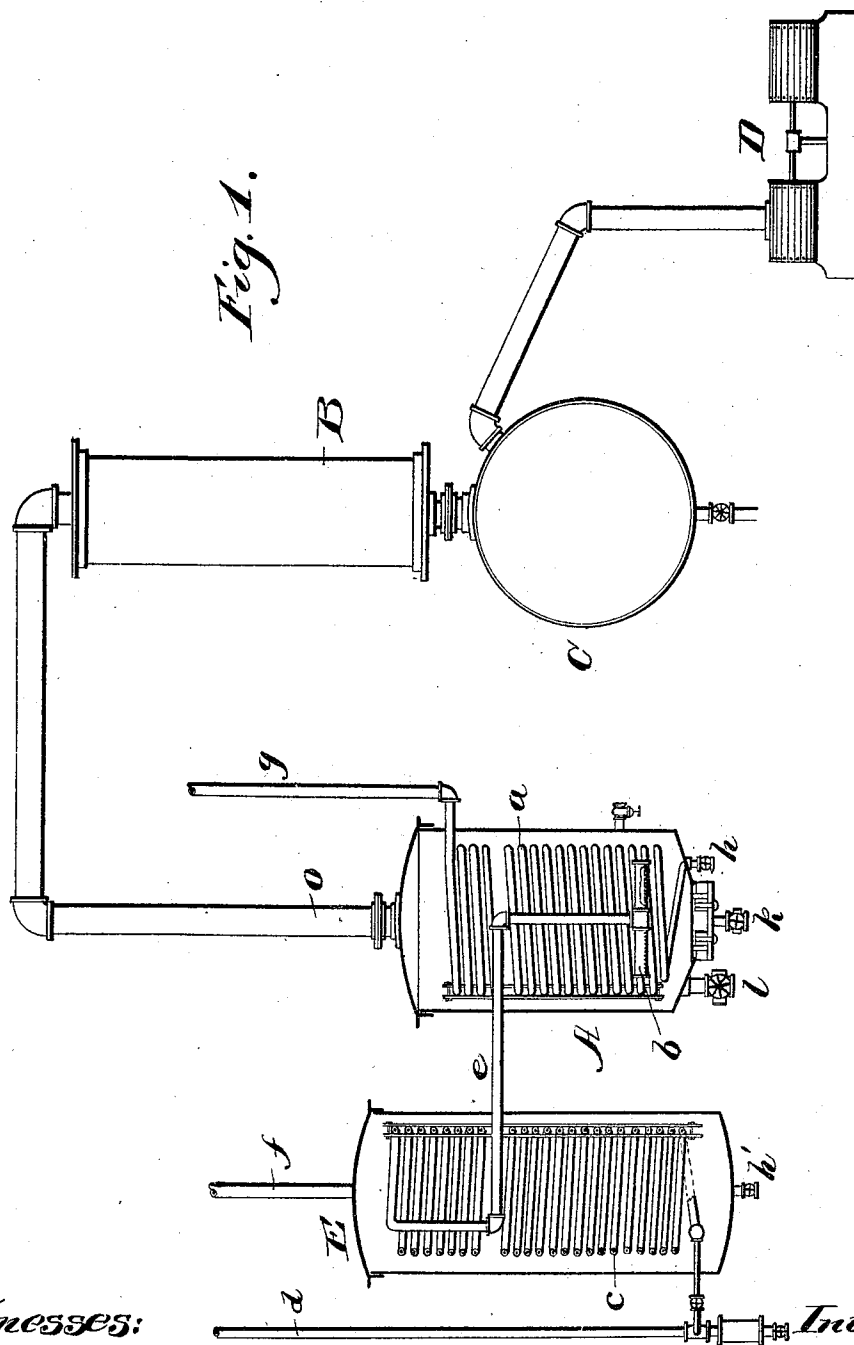
2 Sheets—Sheet 1.

J. VAN RUYMBEKE.

PROCESS OF AND APPARATUS FOR DISTILLING GLYCERIN.

No. 522,132.

Patented June 26, 1894.



Witnesses:

J. B. McGirr.
Wm. M. Kearthy

Inventor.

Joseph Van Ruymbeke
by Frank L. Roper
Attorney

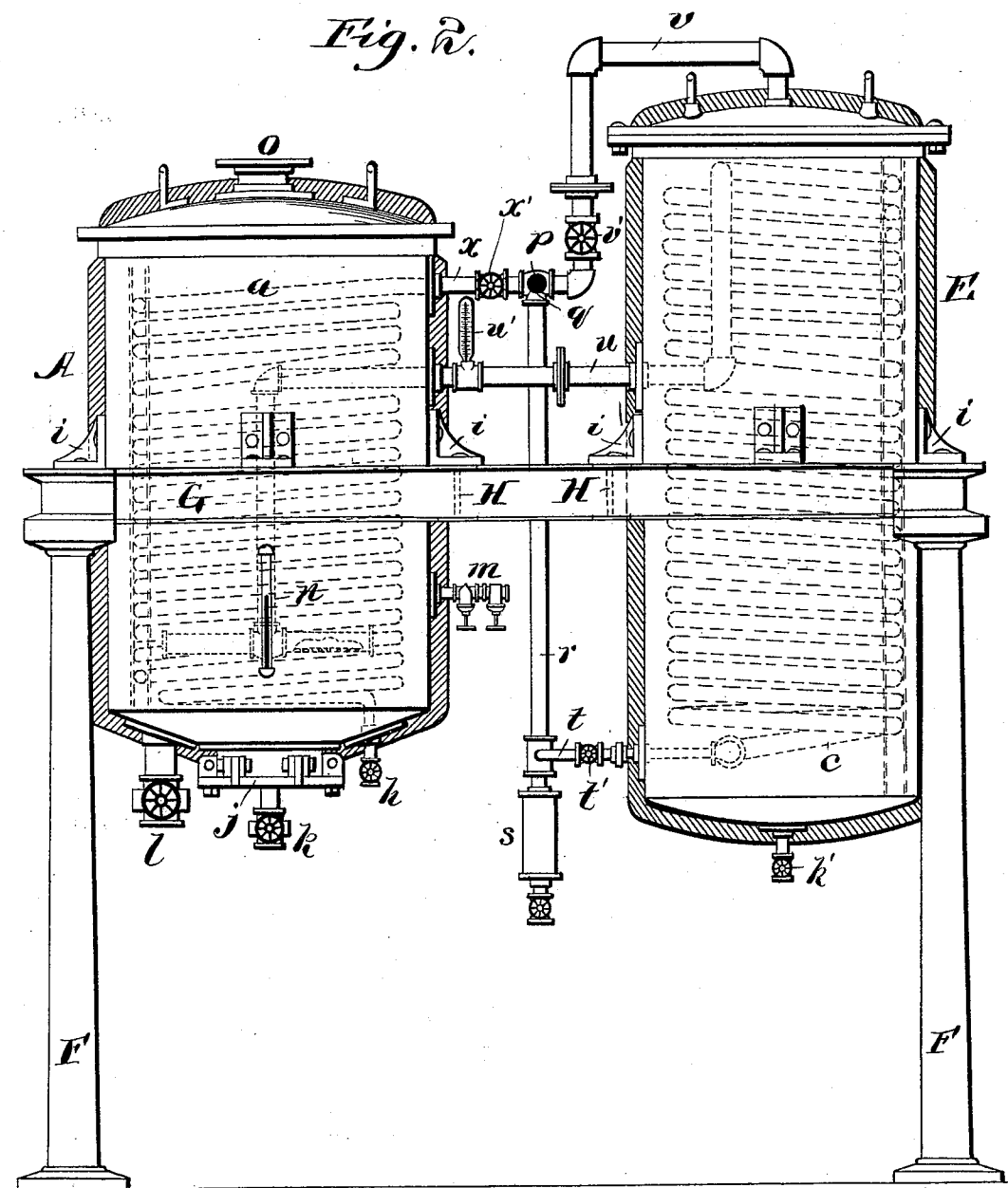
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Fig. 2.



Witnesses:

J. B. McGirr.
Wm. M. McCarthy

Inventor

Joseph Van Ruymbeke
by *Francis L. Roper*
Attorney

UNITED STATES PATENT OFFICE.

JOSEPH VAN RUYMBEKE, OF CHICAGO, ILLINOIS, ASSIGNOR OF ONE-HALF
TO WILLIAM F. JOBBINS, OF SAME PLACE.

PROCESS OF AND APPARATUS FOR DISTILLING GLYCERIN.

SPECIFICATION forming part of Letters Patent No. 522,132, dated June 26, 1894.

Application filed April 23, 1894. Serial No. 508,694. (No model.)

To all whom it may concern:

Be it known that I, JOSEPH VAN RUYMBEKE, a subject of the King of Belgium, residing at Chicago, in the county of Cook and State of Illinois, have invented certain new and useful Improvements in Processes of and Apparatus for Distilling Glycerin and Similar Materials; and I do hereby declare the following to be a full, clear, and exact description of the invention, which will enable others skilled in the art to which it appertains to make and use the same.

My invention relates to a new process of distillation of easily decomposable material, and to apparatus therefor. I shall describe this process as it is carried out in the distillation of glycerin, although it is to be understood that it can also be used in distilling other materials liable to be decomposed during distillation, such as oils, fatty acids, &c.

I shall also describe and claim herein a convenient form of apparatus by means of which my said improved process may be carried out; but I do not restrict myself to said apparatus or to any apparatus for carrying out the same.

In the accompanying drawings: Figure 1, is a diagrammatic view of a portion of a distilling plant, for the purpose of illustrating my process, and Fig. 2, a side elevation of a convenient form of still and reheater in which the novel steps of my process can be carried out.

In both of the above views corresponding parts are designated by the same letters of reference.

Referring to Fig. 1, A represents a still; *a*, is a heating coil in the still; *b*, is an annular distributing pipe, perforated on its under side for injecting steam into the still, such steam being prepared as I shall presently point out; B, is a condenser; C, is a receiving tank for the condensed distillate; D, is a vacuum pump; E, is a heating cylinder preferably adjacent to the still; *c*, is a steam coil within said heating cylinder, which I shall hereinafter refer to as the expansion coil; *d*, is a pipe through which steam is admitted to the expansion coil, and which should either be of smaller diameter than the expansion coil, or else, should be provided with a valve so that the inlet opening into the expansion coil may

be made less than the outlet opening of the said coil, and may have any desired relation to the latter opening so as to make it possible to obtain any desired expansion of the steam from the boiler; *e*, is a pipe which connects the upper end of the expansion coil with the distributing pipe *b*; *f*, is a pipe which connects the heating cylinder E, with any suitable source of steam supply, such as the steam boiler which supplies the injected steam; *g*, is a pipe which connects said boiler or other source of steam supply with one end of the coil *a*; *h*, is a pipe connected with the other end of the coil *a*, for carrying off water of condensation from said coil, or, when there is no appreciable condensation, for carrying off steam from said coil to any point desired; and, *h'*, is a drip pipe for the heating cylinder E.

In Fig. 2, F, F, are heavy standards, four of which are used; G, G, are heavy metal braces bolted to the upper ends of said standards, and H, H, are cross braces, such braces being generally made of ordinary I irons. The still A is supported on said braces G, H, by means of brackets *i*; and the heating cylinder E, is also supported by means of similar brackets at the side of the still. Both the still A, and heating cylinder E are made of boiler iron or steel of sufficient thickness to withstand high pressures.

At the bottom of the still A, is a heavy metal door *j*, adapted to be bolted in place and packed tightly to prevent leaking. The said door *j*, is provided with a draw off pipe *k*, and a larger draw off pipe *l*, is generally provided at one side of the said door. Both of said draw off pipes *k*, *l*, are provided with gate valves therein, and by means of said pipes the residue in the still can be drawn off. The door *j*, is used for the purpose of allowing the interior of the still to be reached for the purpose of cleaning, &c. A pipe *m*, at the side of the still enables the material which is to be distilled to be introduced therein, said pipe being provided with gate valves which are to be closed when a sufficient quantity of material has been introduced into the still. A water column *n*, at the side of the still enables the amount of material therein to be readily determined. At the top of the

still extends the eduction pipe *o*, which leads to the condenser.

p, is a pipe which connects with a boiler or any other suitable source of steam supply, and, *q* is a four-way casting at the end of said pipe. Extending down from the casting *q*, midway between the still *A*, and the reheater *E*, is a steam pipe *r*, provided with an enlarged condensation chamber *s*, at its lower end for receiving any water of condensation which may accumulate in the steam pipes *p* or *r*. The condensation chamber is provided with a drip pipe, at its lower end, by means of which the water or condensation may be removed therefrom. Extending out from the steam pipe *r*, directly above the condensation chamber *s*, and entering the heating cylinder *E*, is a steam pipe *t*, which connects with the lower end of the expansion coil *c*, and which is of smaller diameter than the pipe of said expansion coil, in order that steam in passing from said steam pipe *t* into the expansion coil *c*, will rapidly expand, its temperature being correspondingly diminished, as will be understood. The object of allowing this steam to expand in this way will be presently pointed out.

The steam pipe *t*, is provided with a valve *t'*, therein, by means of which the amount of steam passing through said pipe may be regulated. It is obvious that the pipe *t*, may be of the same size as, or even larger than the steam coil *c*, and that the valve *t'* may yet be so adjusted as to allow the steam to expand in the steam coil as explained. The upper end of the expansion coil connects with a pipe *u*, which passes out through the wall of the heating cylinder *E*, and enters the still *A*, being connected at its lower end to the annular distributing pipe *b*, preferably perforated on its under side as shown. In practice it is desirable that the pipe *u* should be of the same or of a larger diameter than the expansion coil so that the steam from the expansion coil may pass freely through the same. Pipe *u*, is generally provided with a thermometer *u'*, between the still *A*, and the heating cylinder *E*, where it may be readily inspected and by means of which the temperature of the injected steam can be determined. Extending out from one of the horizontal portions of the casting *q*, is a pipe *v*, which leads into the top of the heating cylinder *E*, and by means of which the said heating cylinder may be supplied with steam from the boiler. The said pipe *v*, is provided with a valve *v'* therein, by means of which the supply of steam to the heating cylinder can be regulated. A drip pipe *h'*, at the lower end of the reheater is provided to draw off any accumulated water of condensation. Extending out from the casting *q*, is a pipe *x*, which passes into the still, and connects with the steam coil *a*, therein, such pipe being provided with a valve *x'* therein, by means of which the supply of steam to the coil *a*, can be readily regulated. Any water of condensation

which may accumulate in the coil *a*, may be removed therefrom, through the drip pipe *h*, at the lower end thereof.

In carrying out my process I proceed substantially as follows, attention being particularly directed to Fig. 1: The material to be treated, which is either partly refined glycerin, or a saturated solution of common salt in glycerin, is placed in the still *A* so as to fill the still to about one-third of its capacity. Steam is now introduced through the pipe *f*, and passes into and heats the heating cylinder *E* from which the water of condensation is conducted away by means of the drip pipe *h'*. Steam from the boiler is also passed through the pipe *g*, and steam coil *a*, by means of which the material is independently heated during the process of distillation. The steam from the boiler has of course a temperature above that of the boiling point of the material being distilled. To make distillation more rapid and to lower the temperature of distillation a vacuum of about twenty-eight inches should be maintained in the still by the vacuum pump *D*. Free steam preferably from the same boiler is introduced through the pipe *d*, into the expansion coil *c*, and by reason of the large size of the latter, this steam will rapidly expand and will consequently suffer a diminution in temperature. Since, however, the expansion coil *c*, is heated by free steam in the cylinder *E*, the expanded steam in said coil will be reheated to about the temperature of such free steam, or slightly below such temperature. It is obvious that the free steam may be allowed to expand directly into the heating cylinder *E*, and that it may be reheated by confined steam in a coil within said cylinder. It is further evident that other means for reheating the steam after expansion has taken place, may be used, such as by a steam jacket surrounding the heating cylinder *E*, or by hot air, or by means of a bath of molten metal having a low melting point, like lead; but in any case it is desirable that the means of reheating the free steam should be such as will prevent material variations in temperature. From the expansion coil *c*, the steam which has been thus expanded and then reheated passes through the pipe *e*, and is injected into the material through the perforated distributing pipe *b*. This injection of expanded and reheated steam, will rapidly distill the material, and will carry the distillate over to the condenser *B*, where it condenses and is caught by the receiving tank *C*, from which it may be removed and concentrated.

The operation of the preferable form of still and reheater shown in Fig. 2, is substantially the same as that described, and will be readily understood. Steam from the boiler and under boiler pressure is conveyed from the pipe *p* into the pipes *r*, *v*, and *x*. The steam from the pipe *r*, passes through the small steam pipe *t*, and thence into the expansion coil *c*, where it rapidly expands as before ex-

plained owing to the large size of the latter. The diminution in temperature of this expanded steam is practically regained by heat absorbed from the free steam within the heating cylinder E, conveyed thereto by the pipe v. The amount of steam supplied to the heating cylinder may be adjusted by the valve v', and should be sufficient to reheat the proper amount of expanded steam up to the temperature desired for distillation.

Steam from the pipe x, enters the steam coil a, and heats the liquid in the still during the distillation.

The particular advantage which properly expanded and reheated steam possesses in distillation is, that the steam expands but slightly and consequently causes but slight absorption of heat due to such expansion. When on the other hand, ordinary free steam is used for distillation, it always expands very much in the still, and absorbs heat from the contents of the still, so that distillation is retarded not only because of the diminution in temperature thus produced in the material, but also because of the varying quantities of condensed steam retained by the material.

With super-heated steam, that is to say, steam which has been heated to a high temperature by fire heat or by other means, the same objections are found, and although the diminution in temperature suffered by super-heated steam in expanding in the still, whether a vacuum is used or not, may not be sufficient to cause condensation, yet such steam is undesirable for the distillation of glycerin or similar materials, since decomposition products will be inevitably formed.

The glycerin prepared as above described may after concentration be again distilled to eliminate the last traces of salts.

Although to obtain the best results, the operation should be carried on in a vacuum, and with means of heating independent of the injected expanded and reheated steam, yet to effect the distillation with the omission of either vacuum or the independent means of heating, or both, is also within the scope of my invention, the essence of which consists in the injection into the glycerin of expanded and reheated steam.

Although the expansion of the steam injected may be varied within a very wide limit, I have found that steam coming from the boiler at a pressure of from seventy to one hundred and twenty pounds, when expanded to from twenty to thirty times its original volume, will give excellent results.

Although a slight expansion and reheating of the steam to be injected is beneficial *pro tanto*, still it is preferable that the injected steam be expanded until it has a pressure less than that of saturated steam at the boiling point of the liquid from which the glycerin is distilled. To obtain the best results, it is also necessary that the temperature of the injected steam be kept strictly below the point

at which decomposition products are formed. When a high vacuum is used, the pressure of the injected steam may be less than atmospheric pressure.

Having now described my invention, what I claim is—

1. In the art of distilling glycerin and similar liquids the improvement which consists in injecting into the liquid in the presence of a vacuum, steam at less than atmospheric pressure and at a temperature sufficient to effect distillation, substantially as and for the purpose set forth.

2. In the art of distilling glycerin or similar liquids, the improvement which consists in maintaining the liquid to be distilled at a high vacuum, and in injecting expanded and reheated steam into the liquid, as and for the purpose described.

3. In the art of distilling glycerin or similar liquids, the improvement which consists in maintaining the liquid to be distilled at a high vacuum, in independently heating the liquid, and in injecting expanded and reheated steam into the independently heated liquid, maintained at distilling temperature, as and for the purpose described.

4. In the art of distilling glycerin or similar liquids the improvement which consists in maintaining the liquid to be distilled at a high vacuum, in heating the liquid by means of confined steam, and, in injecting expanded and reheated steam into the heated liquid, maintained at distilling temperature, as and for the purpose described.

5. In the art of distilling glycerin or similar materials, the improvements which consist in maintaining the liquid to be distilled at a high vacuum, in heating the liquid by means of confined steam, in expanding free steam before entering the still, in reheating such expanded steam by confined live steam of the same temperature as the expanded steam previous to expansion, so as to compensate for reduction of temperature caused by the expansion, and in injecting the expanded and reheated steam into the heated liquid maintained at distilling temperature, as and for the purpose described.

6. A distilling apparatus, consisting of a still, an expansion coil, the inlet opening of which is smaller than the outlet opening, a heating cylinder for the expansion coil, and a perforated delivery pipe in the still, and connected with the expansion coil, substantially as described.

7. A distilling apparatus, consisting of a still, a vacuum pump connected with the still, an expansion coil, the inlet opening of which is smaller than the outlet opening, a heating cylinder for the expansion coil, and a perforated delivery pipe in the still and connected with the expansion coil, substantially as described.

8. A distilling apparatus, consisting of a still, a heating coil in the still, an expansion coil, the inlet opening of which is smaller than

the outlet opening, a heating cylinder for the expansion coil, and a perforated delivery pipe in the still and connected with the expansion coil, substantially as described.

5 9. A distilling apparatus, consisting of a still, a vacuum pump connected therewith, a heating coil for heating the contents of the still, an expansion coil connected with the still, and with a source of steam supply, the
10 inlet opening of said expansion coil being smaller than the outlet opening, and a heating cylinder for reheating the steam in the expansion coil, substantially as described.

10. A distilling apparatus, consisting of a
15 still, a vacuum pump connected therewith; means for heating the contents of the still; an expansion coil connected with the still and with the boiler, the inlet opening of said expansion coil being smaller than the outlet
20 opening, and a reheater for heating the expansion coil, substantially as described.

11. A distilling apparatus, consisting of a still, a vacuum pump connected therewith, a heating cylinder, an expansion coil in said
25 heating cylinder and connected with the still, the inlet opening of said expansion coil being smaller than the outlet opening, and sources of supply for steam in the said heating cylinder and expansion coil, substantially as de-
30 scribed.

12. A distilling apparatus, consisting of a still, a vacuum pump connected therewith, a heating cylinder adjacent to the still and connected with a source of steam supply, a heat-
35 ing coil within the still, and an expansion coil in said heating cylinder and connected with the still, the inlet opening of said expansion coil being smaller than the outlet opening, substantially as described.

40 13. A distilling apparatus, consisting of a still, a vacuum pump connected therewith, a heating cylinder adjacent to the still and connected with a source of steam supply, a heating coil within the still, connected with
45 the same source of steam supply, a distribut-

ing pipe within the still, and an expansion coil within the said heating cylinder and connected with said distributing pipe, the inlet opening of said expansion coil being smaller than the outlet opening, substantially as de- 50 scribed.

14. A distilling apparatus, consisting of a still, a vacuum pump connected therewith, a heating cylinder adjacent to the still, connected with a source of steam supply, a heat- 55 ing coil within the still, connected with the same source of steam supply, and an expansion coil within said heating cylinder, connected with the still and supplied with steam from the same source of steam supply, the
60 inlet opening of said expansion coil being smaller than the outlet opening, substantially as described.

15. In a distilling apparatus, the combination of the standards F, the braces G and H, 65 bolted to the upper end of the same, the still A and heating cylinder E, supported by said braces, the steam coil *a* within said still, and the expansion coil *c*, within said heating cylinder, the inlet opening of said expansion
70 coil being smaller than the outlet opening, substantially as described.

16. In a distilling apparatus, the combination of the standards F, the braces G and H, 75 bolted to the upper end of the same; the still A and heating cylinder E, supported by said braces, the steam coil *a*, within said still, the expansion coil *c* within said heating cylinder, the inlet opening of said expansion coil being smaller than the outlet opening, and a
80 common source of steam supply for said steam coil, expansion coil and heating cylinder, substantially as described.

This specification signed and witnessed the 13th day of April, 1894.

JOSEPH VAN RUYMBEKE.

Witnesses:

N. LONGLUND,
CHAS. H. CHAPIN.