(No Model.) 3 Sheets—Sheet 1.

J. VAN RUYMBEKE.

DISTILLING APPARATUS.

No. 568,219. Patented Sept. 22, 1896.

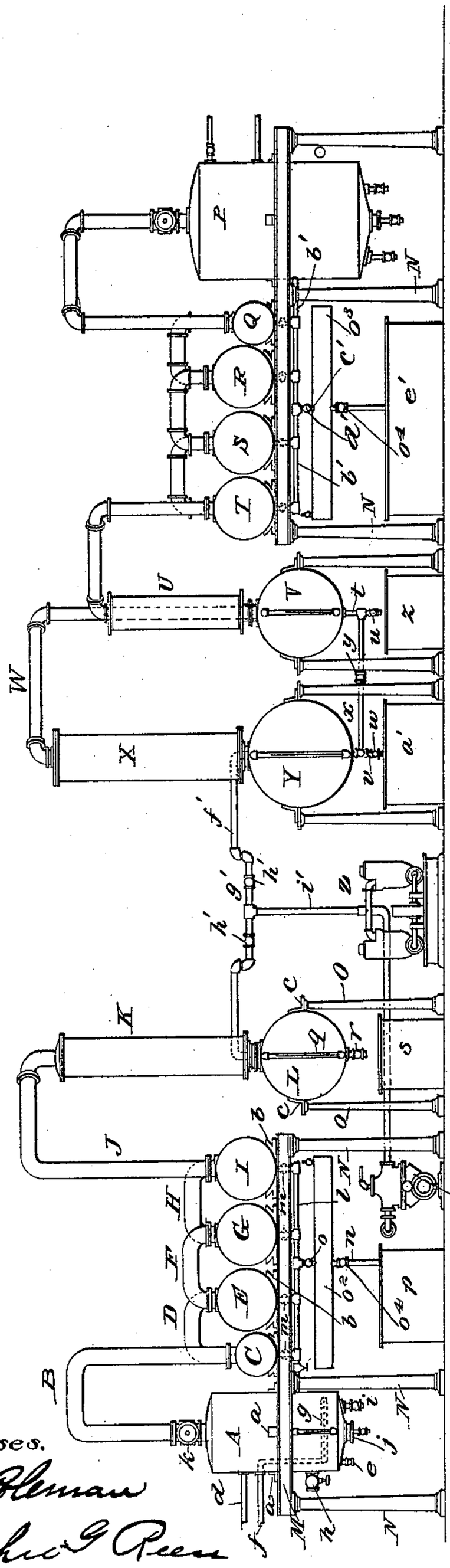

Fig. 1.

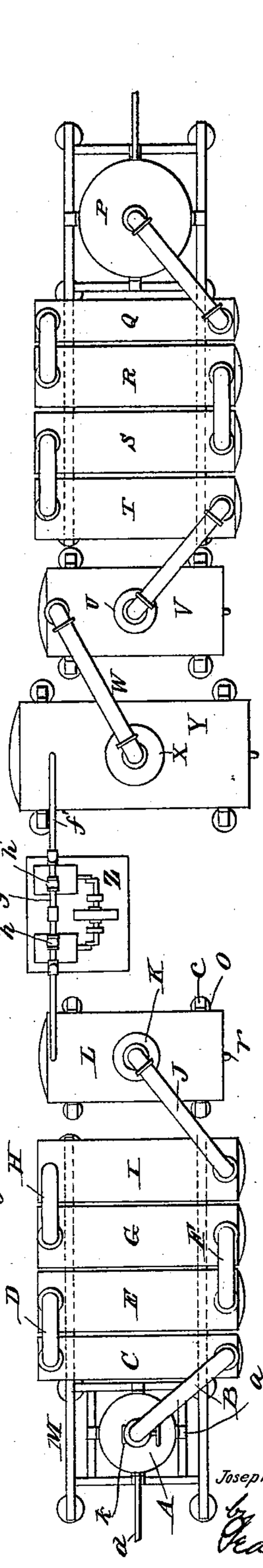

Fig. 2.

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J. VAN RUYMBEKE.

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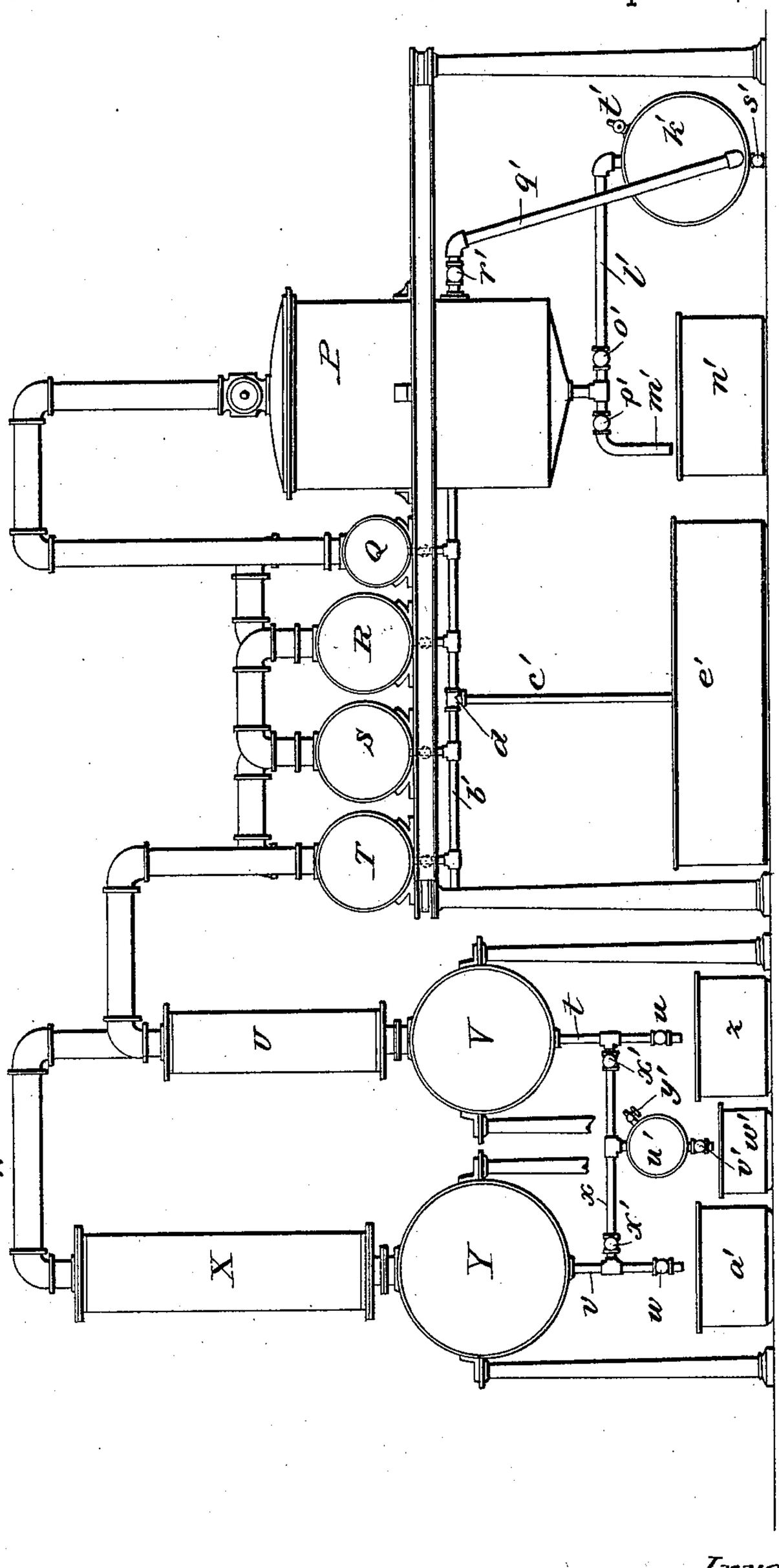

Fig. 3

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(No Model.) 3 Sheets—Sheet 3.

J. VAN RUYMBEKE.

DISTILLING APPARATUS.

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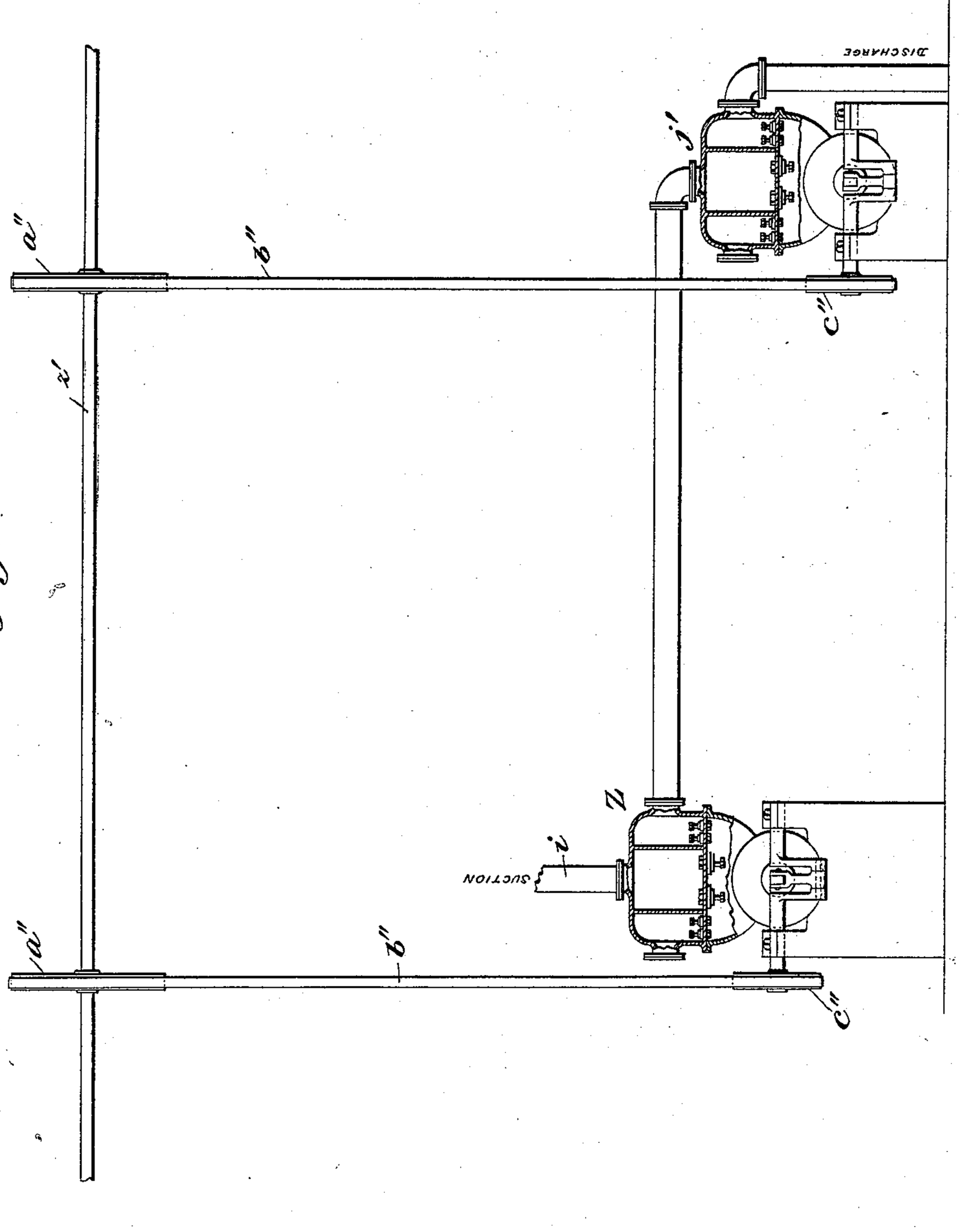

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UNITED STATES PATENT OFFICE.

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

DISTILLING APPARATUS.

SPECIFICATION forming part of Letters Patent No. 568,219, dated September 22, 1896.

Application filed August 16, 1895. Serial No. 559,534. (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, county of Cook, in the State of Illinois, have invented certain new and useful Improvements in Distilling Apparatus; 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 various new and useful improvements in distilling apparatus which is particularly adapted for the distillation of glycerin, but which may be employed for the distillation of other materials.

In obtaining glycerin of high specific gravity and of sufficient purity for commercial purposes from crude glycerin obtained, for instance, from waste soap-lyes, it is preferable to employ two distinct distilling-plants, the sweet water obtained from the first distillation after concentration being again distilled and finally concentrated to the desired specific gravity.

The apparatus constituting my present invention combines two stills for this purpose in very compact and efficient form, as will be more fully explained, and specifically pointed out in the claims.

In the accompanying drawings, Figure 1 represents a side elevation of the apparatus; Fig. 2, a plan view of the same; Fig. 3, an enlarged sectional view, partly in elevation, of the still, separating-drums, condenser, and sweet-water drums forming the right-hand part of the apparatus shown in Fig. 1; and Fig. 4, a diagrammatic view, partly in section, of two valved mechanical vacuum-pumps connected together in accordance with my present invention, illustrating conventional mechanism for operating both of said pumps simultaneously.

In all of the above views corresponding parts are represented by the same letters of reference.

A represents a cylindrical still. B is an eduction-pipe therefrom connected with a separating-drum C.

D is a pipe connecting the separating-drum C with a second separating-drum E, preferably larger in diameter than the drum C.

F is a pipe connecting the separating-drum E with a third separating-drum G.

H is a pipe connecting the separating-drum G with a fourth separating-drum I.

J is a pipe connecting the separating-drum I with a cooler or condenser K, and L is a sweet-water drum beneath the condenser or cooler K and supporting the same for receiving the material condensed therein.

The still A and separating-drums C, E, G, and I are carried on a supporting-frame M, made, preferably, of I-beams, carried at the top of and bolted to columns or pillars N, so as to be elevated a sufficient distance from the floor. The still A is supported within the frame M by means of small brackets *a*, riveted to the sides of the still A and bolted to said frame. The separating-drums C, E, G, and I are supported on said frame by means of cradles *b*, which are bolted to I-beams of said frame. The sweet-water drum L is carried on pillars or columns O by means of brackets *c*, riveted to the sides of said sweet-water drum and bolted to the tops of said pillars or columns.

Viewing the apparatus in the position shown, it will be noted that the eduction-pipe B connects with the separating-drum C near the front of said separating-drum, that the pipe D connects the drums C and E near the back of said separating-drums, that the pipe F connects the drums E and G near the front of said separating-drums, that the pipe H connects the separating-drums G and I near the back of said drums, and that the pipe J connects with the separating-drum I near the front thereof, whereby the vapors from the still A are caused to pass from the eduction-pipe B through the entire lengths of the respective separating-drums, as will be understood.

A steam-pipe *d* connects with a heating-coil which is mounted within the still A for heating the contents thereof, the water condensed in said steam-coil being removed therefrom by a drip-pipe *e*. Instead of heat-

ing the liquid in the still by means of a steam-coil, as explained, other means for this purpose may be employed, such as a steam-jacket.

Steam is injected into the liquid treated for the purpose of distilling the same through a steam-pipe *f*, which connects at its lower end with a perforated injection pipe or jet *g*. The steam injected may be either live steam, superheated steam, expanded or reheated steam, or steam prepared in any suitable way, or, instead of steam, air, gas, or any other vapor may be injected suitable for the distillation of the particular material desired.

The material is introduced into the still A by a valved pipe *h*, and the residuum in the still may be removed therefrom by the valved pipe *i*. A manhole *j* with valved outlet in center at the bottom of the still is employed for cleaning the interior thereof. A gage or water column at the side of the still indicates the height of the liquid therein. The eduction-pipe B may be provided with a valve *k* therein for shutting off the still when desired.

l represents a pipe mounted beneath the separating-drums C, E, G, and I, opening at one end into the still, as shown, and closed at the other end, and *m m m* represent short valved pipes connecting the respective separating-drums with the pipe *l*.

A pipe *n*, having a valve *o* therein, extends down from the pipe 1, and the tank *p* beneath said pipe *n* receives the liquid flowing from the same. The sweet-water drum L is provided on its front end with a gage or water column *q* for indicating the height of the liquid therein, and underneath with a valved draw-off pipe *r* for drawing off the liquid from said sweet-water drum into a tank *s*, placed beneath the same.

The elements which I have above described comprise one of the distilling-plants constituting my present apparatus. The other distillation-plant, comprising the rest of my apparatus and shown at the right-hand side of Fig. 1, consists of the still P, separating-drums Q, R, S, and T, a cooler or condenser U, and a sweet-water drum V, beneath said cooler or condenser and connected with the same. These elements are made preferably in the same way as the corresponding elements before described, and for convenience and economy iron or steel may be employed for their construction.

W is a pipe extending up from the sweet-water drum V and connected with the larger cooler or condenser X, beneath which is a second sweet-water drum Y. The sweet-water drum V is provided with a draw-off pipe *t*, having a valve *u* at its lower end, and the sweet-water drum Y is provided with a similar draw-off pipe *v*, having a valve *w* at its lower end. A pipe *x*, having a valve *y* therein, connects the draw-off pipes *v* and *t* above the valves *u* and *w*, respectively. Receiving-tanks *z* and *a'* are located beneath the draw-off pipes *t* and *v*, as shown.

The separating-drums Q, R, S, and T are connected by short valved pipes to a pipe *b'*, which enters the still P, and *c'* is a pipe having a valve *d'* at its upper end for drawing off the material from any or all of said separating-drums, when desired, into a large receiving-tank *e'*.

The sweet-water drums L and Y are connected together at their rear ends by a pipe *f'*, which pipe is provided at its central part with a depressed portion *g'*, whereby liquids are prevented from flowing from one sweet-water drum to the other. The pipe *f'* is provided with valves *h' h'* therein, and extending down from said pipe between said valves is a small pipe *i'*, which connects with a vacuum-pump Z, which is preferably a dry vacuum-pump.

When a very high vacuum is desired in the apparatus, as is the case when glycerin is being distilled, I prefer to employ a second vacuum-pump *j'*, which connects with the exit-pipe of the vacuum-pump Z, the two pumps being operated together. I find that the second vacuum-pump supplements the first pump very materially, and that by its use the desired high vacuum is produced. Both vacuum-pumps are ordinary commercial mechanical valved vacuum-pumps of any approved type.

In Fig. 4 the connection of the vacuum-pumps is illustrated on a larger scale, the suction-pipe of the pump *j'* being connected to the discharge from the pump Z, so as to enhance the action of the latter pump, as explained. In this figure I illustrate conventional means for operating both of the pumps simultaneously, comprising the horizontal shaft *z'*, carrying pulley-wheels *a''*, which connect by belts *b''* with the drive-wheels *c''* of both pumps.

In Fig. 1 I show receiving-drums o^2 and o^3, connected with the draw-off pipes *n* and *c'* of the two series of separating-drums, respectively, and provided with valves o^4 beneath them. Said receiving-drums are provided with petcocks, as shown, for admitting air therein. The object of these receiving-drums is to allow the contents of the separating-drums to be drawn off without destroying the vacuum in the apparatus, this result being accomplished at the left-hand side of the apparatus, for example, by opening the valve *o*, the valve o^4 being closed, thereby allowing the liquid to flow into the drum o^2, after which the valve *o* is closed and valve o^4 and petcock opened to allow the liquid to flow out of said drum into the tank *p*.

It is obvious that by opening the valve of any one or more of the separating-drums the contents thereof may be removed through the receiving-drum o^2 or o^3 independent of the other separating-drums.

In Fig. 3, which shows the right-hand side of the apparatus on a larger scale, I illustrate additional features, which are of especial importance when the crude glycerin con-

tains glycol or other substances more volatile than glycerin and which it is desired to separate from the glycerin while in the still. At one side of the still and below the same is placed a receiving-drum k' of sufficient capacity for the purpose. The draw-off pipe of the still connects with this drum by a pipe l', having an outlet m' at its other end. A tank n' is placed beneath this outlet m'. Valves o' and p' are placed in the pipe l' on each side of the draw-off pipe of the still. A pipe q', having a valve r', connects the interior of the drum k' with the still. Said drum is provided with a valved draw-off pipe s' and with a petcock t'. In said Fig. 3 I also show a drum u', connected to the pipe x and provided with a valved draw-off pipe v', beneath which is a receiving-tank w'. Valves x' x' are mounted in the pipe x on each side of the drum u', and said drum is preferably provided with a petcock y'. This drum u' is of importance for use in connection with the drum k' when the apparatus is employed in fractionating, as will be explained.

The operation of my complete apparatus for the distillation of crude glycerin is as follows: The material is introduced into the still P, so as to fill the same to about one-third of its capacity, either by pumping the same or by the suction produced by the operation of the vacuum-pumps Z and j'. The vacuum-pumps continue to operate and produce the desired degree of vacuum in the sweet-water drums V and Y, separating-drums T, S, R, and Q, and still P. The material in the still P is independently heated, such as by a steam-coil corresponding to the coil d of the still A, and steam or other vaporizing agent of sufficient temperature to rapidly distil the liquid therein is injected therein. The vapors evolved from the still P pass successively through the separating-drums Q, R, S, and T, thence through the condenser U, sweet-water drum V, pipe W, condenser X, and sweet-water drum Y. The vapors evolved from the still in passing through the separating-drums Q, R, S, and T will be partially condensed therein.

In practice I find that in the first drum Q glycerin with impurities are collected and condensed which sometimes require to be returned to the still P through the pipe b' for redistillation, that in the separating-drums R and S glycerin generally of high specific gravity and of sufficient purity for dynamite purposes is collected and does not require subsequent treatment, and that in the separating-drum T glycerin with varying quantities of water are condensed, which may therefore require concentration. The vapors passing through the condensers U and X, respectively, are condensed therein. The resulting sweet waters, consisting of condensed steam carrying from one per cent. to ten per cent. or more of glycerin, are collected in the drums V and Y. By employing two or more separate condensers U and X, as explained, any condensable vapors are caught in the apparatus and prevented from passing through the vacuum-pump Z. The sweet waters collected in the drums V and Y are removed therefrom into the tanks a' or z, and the dilute glycerin caught in the separating-drum T is removed therefrom through the pipe b' and c', and these products are then concentrated. The glycerin collected in the separating-drums R and S, as stated, is generally of sufficient purity for dynamite purposes, and therefore may not need to be further treated, although when great purity is desired it may be redistilled alone or with the concentrated sweet waters. These concentrated sweet waters are distilled in the still A, being introduced therein preferably by means of the suction produced by the vacuum-pumps.

It will be understood that both distilling-plants may be operated at the same time, the vacuum in each being maintained by the vacuum pump or pumps. The concentrated sweet waters introduced into the still A are now maintained at a temperature approximating to their boiling-point, and steam or other vaporizing agent is introduced of sufficient temperature for the purpose through the injection-pipe g, so as to rapidly distil the material.

Products from the still A pass through the pipe B into the separating-drum C, in which glycerin and the impurities, if any, carried over with the vapors are caught. From this drum the products pass through the pipe D into the separating-drum E, thence through the pipe F into the separating-drum G. In these drums glycerin generally of great purity and excellent quality is collected, the temperature maintained in said drums being sufficient to prevent condensation of almost no water therein.

From the drum G the products pass through the pipe H into the separating-drum I, the temperature of which is generally sufficiently low to cause water to condense, more or less, with glycerin, and which may therefore require to be concentrated. From the separating-drum I the products, which now consist of water carrying varying proportions of glycerin, pass into the condenser or cooler K, by which the remaining water and glycerin is condensed and collected in the sweet-water drum L.

The dilute glycerin separated in the drum I and the sweet water condensed and collected in the drum L are now concentrated in a copper or other suitable concentrating apparatus to the desired specific gravity, the resulting product being glycerin of great purity. The glycerin separated in the drums E and G, as stated, is generally of an excellent quality, and requires no concentration. It will therefore be seen that I have produced an apparatus particularly designed for the distillation of glycerin from crude glycerin which is simple in construction, is very com-

pact, efficient in use, and wherein a single vacuum-pump or other vacuum device is required.

The products condensed and collected in the drum V consist of water with varying proportions of glycerin, and ordinarily any products which pass through the pipe W and condenser X into the drum Y carry little or no glycerin, but consist almost entirely or wholly of condensed steam. This water may therefore be removed from the drum Y into the tank a' and allowed to run to waste, but if upon test it is found that the products condensed in the drum Y contain glycerin it may be allowed to flow into the tank z through the pipe x, the valve w being closed.

I have found in practice that crude glycerin obtained from the waste soap-lyes of some manufacturers contains varying quantities of glycol, which is more volatile than the glycerin, and should be separated therefrom. By means of the additional appliances shown in Fig. 3 I am enabled to use my apparatus as a fractionating-plant for the separation of the glycol from the glycerin and for the subsequent distillation of the glycerin. These operations are carried out as follows: The crude glycerin is placed in the still P, as heretofore, and the valves in the draw-off pipes of the drums Q, R, S, and T are left open, the other valves being closed. The vacuum-pump being started, steam is injected into the liquid and the liquid is preferably independently heated by a steam-coil.

The temperature of the liquid in the still is maintained above the distilling-point of the glycol, but below that of the glycerin, so that the glycol distils off and passes through the various separating-drums, being collected in the drums V and Y. The objects of keeping the valves in the draw-off pipes of the separating-drums open are to allow said drums to be heated to a sufficiently high temperature so that after awhile no glycol can condense and accumulate therein, and also to allow any glycerin which may be mechanically carried over with the glycol to be intercepted by said separating-drums and flow back into the still. This operation is continued until all the glycol has distilled off, whereupon the valve o' is opened and the glycerin from the still runs into the drum k'.

The still is again partially filled with crude glycerin, and the operations above described are repeated until the drum k' is entirely full. The glycol condensed in the drums V and Y is removed therefrom from time to time and flows into the drum u', the valves x' x' being opened and the valves u and w being closed; or, instead, said valves may be left continuously opened or closed, as required, so that the glycol will accumulate continuously in the drum u'. When the drum u' is to be emptied, the valves x' x' are closed and the valve v' and petcock y' opened, so that the liquid flows out of said drum without affecting the vacuum in the apparatus. The glycerin which has accumulated in the drum k' is now drawn therefrom into the still by suction through the pipe q', the valve r' and petcock t' being opened, and the temperature of the liquid increases, so that the glycerin will be distilled in the way I have described above. Any foots in the still are preferably removed through the outlet-pipe m', although, if it is desired to keep the vacuum unbroken, the foots may be drawn off into the drum k' and thence removed through the pipe s'. By these means I am enabled to employ my improved apparatus as a fractionating-plant, and the distillates produced may be separately removed therefrom without destroying the vacuum therein.

Although I have above described the use of four separating-drums with each still, it is of course to be understood that a greater or lesser number of such drums may be employed.

Having now described my invention, what I claim as new, and desire to secure by Letters Patent, is as follows:

1. In a distillation or other vacuum apparatus, the combination with a closed vessel, of a vacuum-pump connected therewith, and a second valved mechanical vacuum-pump connected with and supplementing the action of the first valved mechanical vacuum-pump, substantially as set forth.

2. In a distillation or other vacuum apparatus, the combination with two closed vessels, of a valved mechanical vacuum-pump common to both vessels for maintaining a vacuum therein, and a second valved mechanical vacuum-pump connected with and supplementing the action of the first vacuum-pump, substantially as set forth.

3. In a vacuum apparatus for distilling glycerin and other decomposable materials, the combination of a closed vessel for containing the material, an injection-pipe for injecting steam into the material for the purpose of distilling the same, a mechanical valved vacuum-pump connected with the closed vessel for constantly maintaining a vacuum therein, a second mechanical valved vacuum-pump connected with the exit of the first pump and supplementing the action of the same, whereby the valves of both of said pumps will be interposed between the atmosphere and the closed vessel, and means for constantly and simultaneously operating both of said mechanical pumps, substantially as set forth.

4. In a vacuum apparatus for distilling glycerin and other decomposable materials, the combination of two closed vessels for containing the material, an injection-pipe in each vessel for injecting steam into the material therein for the purpose of distilling the same, a mechanical valved vacuum-pump connected with both of said vessels for constantly maintaining a vacuum therein, a second mechanical valved vacuum-pump con-

nected with the exit of the first pump and supplementing the action of the same, whereby the valves of both of said pumps will be interposed between the atmosphere and the closed vessel, and means for constantly and simultaneously operating both of said mechanical pumps, substantially as set forth.

5. In an apparatus for the distillation of glycerin and other materials, the combination with the still P, separating-drums Q, R, S and T, connected together and with said still, cooler or condenser U, and sweet-water drum V, of the still A, separating-drums C, E, G and I, connected together and with said still, cooler or condenser K, and sweet-water drum L, and a vacuum-pump Z connected with the sweet-water drums L and Y, for maintaining a vacuum in the stills A and P, substantially as set forth.

6. In an apparatus for the distillation of glycerin and other materials, the combination with the still P, separating-drums Q, R, S and T, connected together and with said still, cooler or condenser U, and sweet-water drum V, of the still A, separating-drums C, E, G and I, connected together and with said still, cooler or condenser K, and sweet-water drum L, a pipe f' communicating with the sweet-water drums L and Y, a depressed section g' in said pipe, valves h', h', therein, and a vacuum-pump Z connected to said pipe f' between said valves, substantially as set forth.

7. In an apparatus for the distillation of glycerin and other materials, the combination of a still, a condenser connected with said still, a sweet-water drum beneath said condenser, a second condenser connected with said sweet-water drum, and a second sweet-water drum beneath said second condenser, substantially as set forth.

8. In an apparatus for the distillation of glycerin and other materials, the combination of a still P, a condenser U connected with said still, a sweet-water drum V connected with said condenser U, a draw-off pipe t for said drum, a tank z beneath said draw-off pipe, a condenser X connected with said drum V, a sweet-water drum Y connected with said condenser X, a draw-off pipe v for said drum Y, and a valved pipe x connecting said draw-off pipes t and v, substantially as set forth.

9. In an apparatus for the distillation of glycerin and other materials, the combination of a still P, a condenser U connected with said still, a sweet-water drum V connected with said condenser U, a draw-off pipe t for said drum, a condenser X connected with said drum V, a sweet-water drum Y connected with said condenser X, a draw-off pipe v for said drum Y, a valved pipe x connecting said draw-off pipes t and v, and a drum u' connected with said pipe x, substantially as set forth.

10. In an apparatus for the distillation of glycerin and other materials, the combination of a still P, a series of connected separating-drums for said still, a condenser U connected with said separating-drums, a sweet-water drum V connected with said condenser U, a draw-off pipe t for said drum, a tank z beneath said draw-off pipe, a condenser X connected with said drum V, a sweet-water drum Y connected with said condenser X, a draw-off pipe v for said drum Y, and a valved pipe x connecting said draw-off pipes t and v, substantially as set forth.

11. In an apparatus for the distillation of glycerin and other materials, the combination of a still P, a series of connected separating-drums for said still, a condenser U connected with said separating-drums, a sweet-water drum V connected with said condenser U, a draw-off pipe t for said drum, a tank z beneath said draw-off pipe, a condenser X connected with said drum V, a sweet-water drum Y connected with said condenser X, a draw-off pipe v for said drum Y, a tank a' beneath said draw-off pipe, and a valved pipe x connecting said draw-off pipes t and v, substantially as set forth.

12. In a vacuum apparatus for the distillation of gycerin and other materials, the combination with a still and draw-off pipe therefor, of a closed drum adjacent to the still and connected with said draw-off pipe, and a valved pipe connecting said drum with the still, substantially as set forth.

13. In a vacuum apparatus for the distillation of glycerin and other materials, the combination with a still and draw-off pipe therefor, of a closed drum adjacent to the still and connected with said draw-off pipe, and a valved pipe connected with the still and extending inside of said drum, substantially as set forth.

14. In a vacuum apparatus for the distillation of glycerin and other materials, the combination with a still and draw-off pipe therefor, of a closed drum adjacent to the still and connected with said draw-off pipe, a valved pipe connected with the still, and extending into said drum, and a petcock on said drum, substantially as set forth.

15. In a vacuum apparatus for the distillation of glycerin and other materials, the combination with a still and draw-off pipe therefor, of a closed drum adjacent to the still and connected with said draw-off pipe, a valved pipe connected with the still at a point above the bottom thereof and extending into said drum, and a pipe connecting the bottom of said still with said drum, substantially as set forth.

16. In a vacuum apparatus for the distillation of glycerin and other materials, the combination with a still and draw-off pipe therefor, of a closed drum adjacent to the still and connected with said draw-off pipe, a valved pipe connected with the still at a point above the bottom thereof and extending into said

drum, a pipe connecting the bottom of said still with said drum, and a petcock on said drum, substantially as set forth.

17. In an apparatus for the distillation of glycerin and other materials, the combination with a still of a series of separating-drums connected with said still and with each other, a draw-off pipe for each separating-drum, a pipe connecting said draw-off pipes, a pipe communicating with the latter pipe, a closed drum in said pipe, and valves in said pipe above and below said closed drum, substantially as set forth.

This specification signed and witnessed the 18th day of May, 1895.

JOSEPH VAN RUYMBEKE.

Witnesses:
THOS. M. STARKE,
JAMES MCINTYRE.