

(No Model.)

4 Sheets—Sheet 1.

A. DOMEIER & O. C. HAGEMANN.

APPARATUS FOR THE DISTILLATION OF CONCENTRATED SOAP LYE.

No. 428,470.

Patented May 20, 1890.

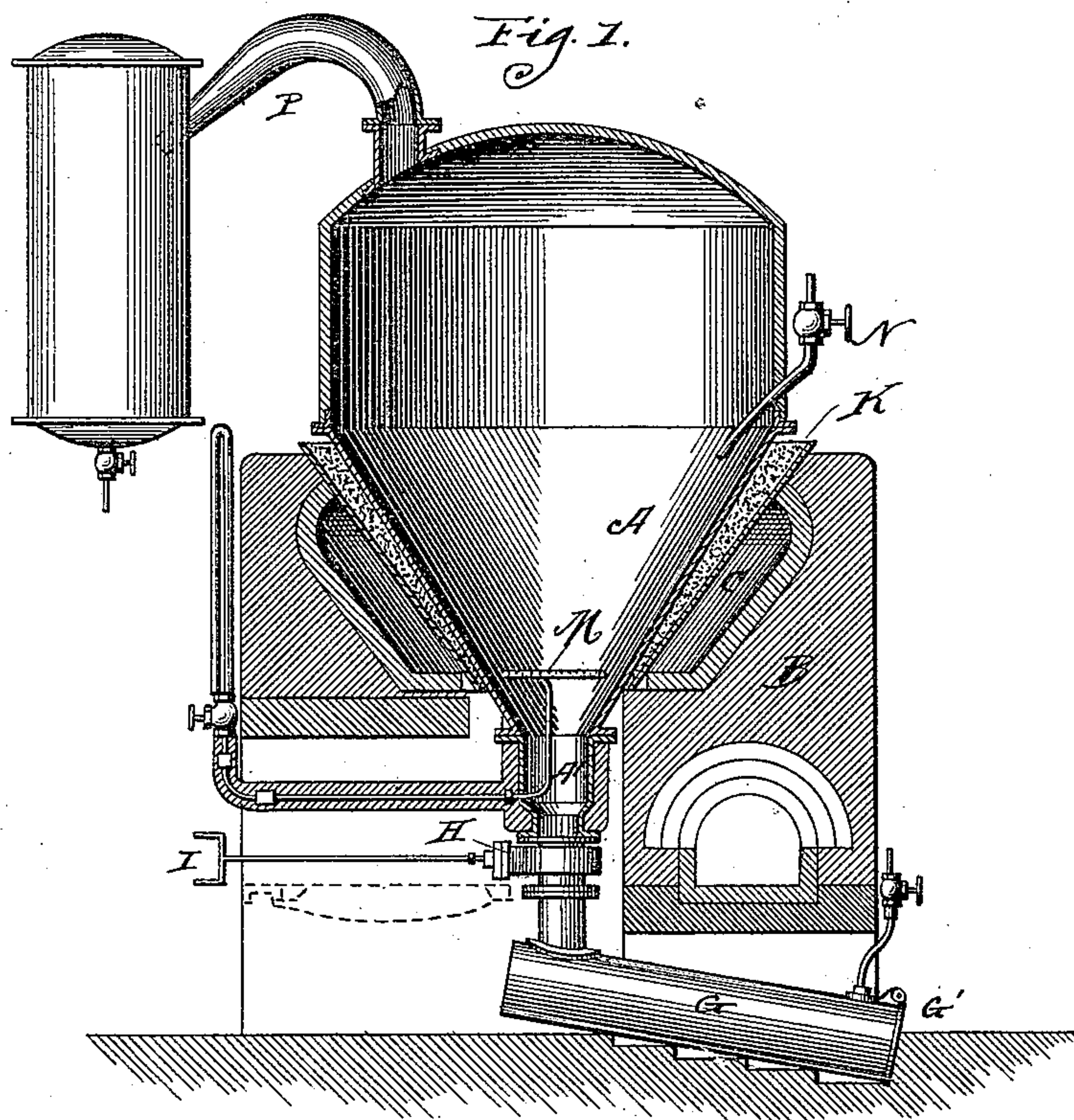
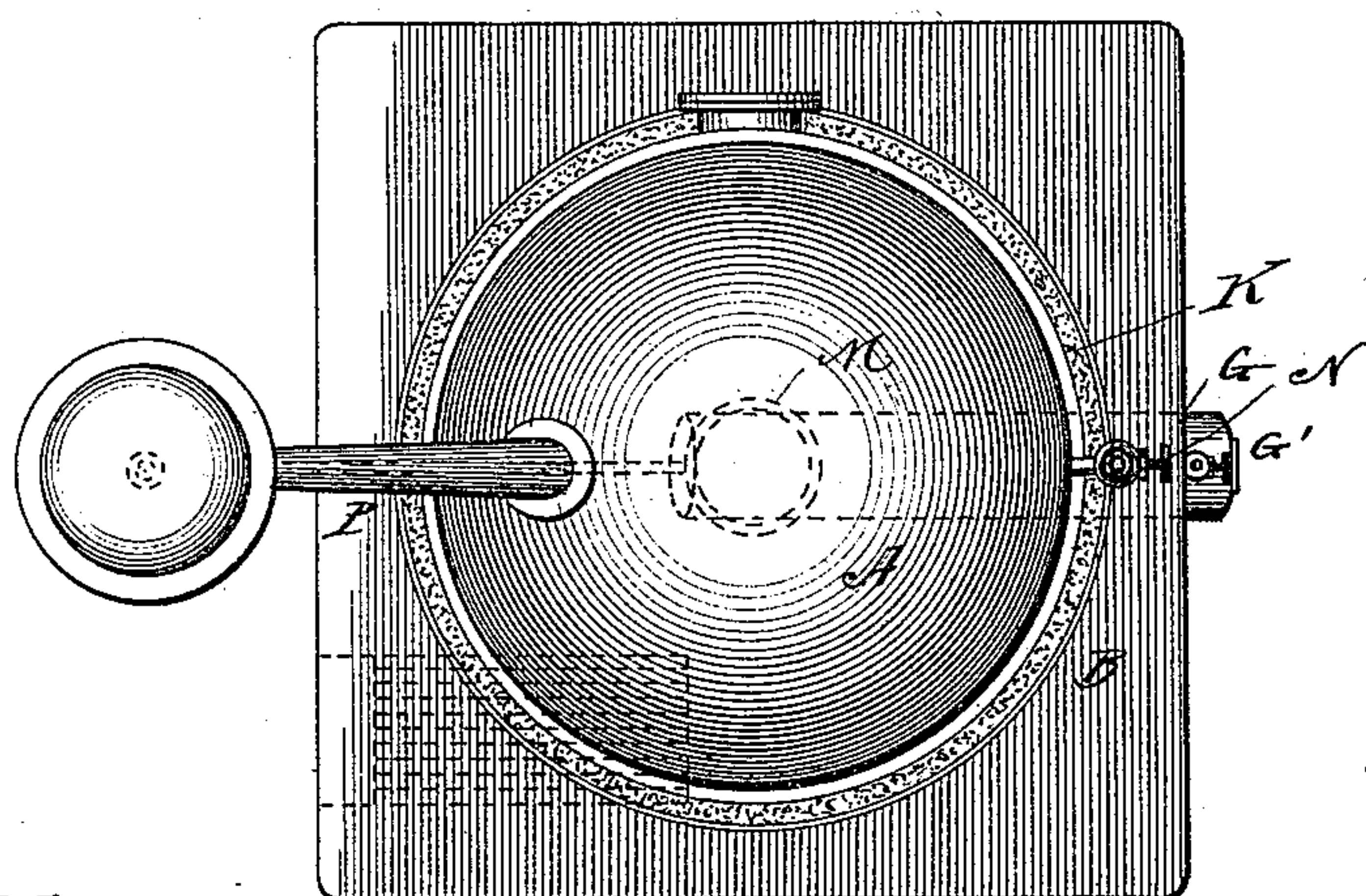


Fig. 2.



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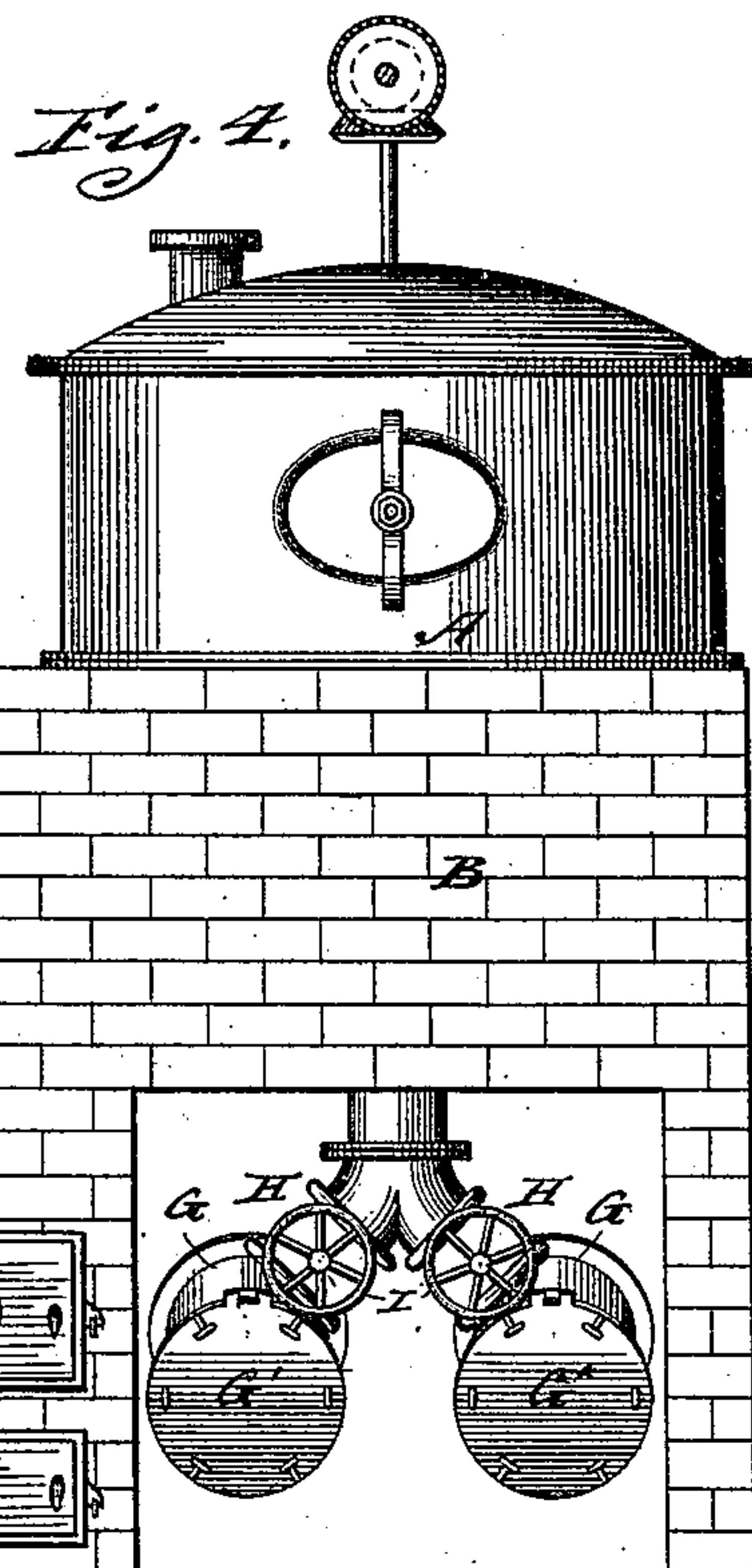
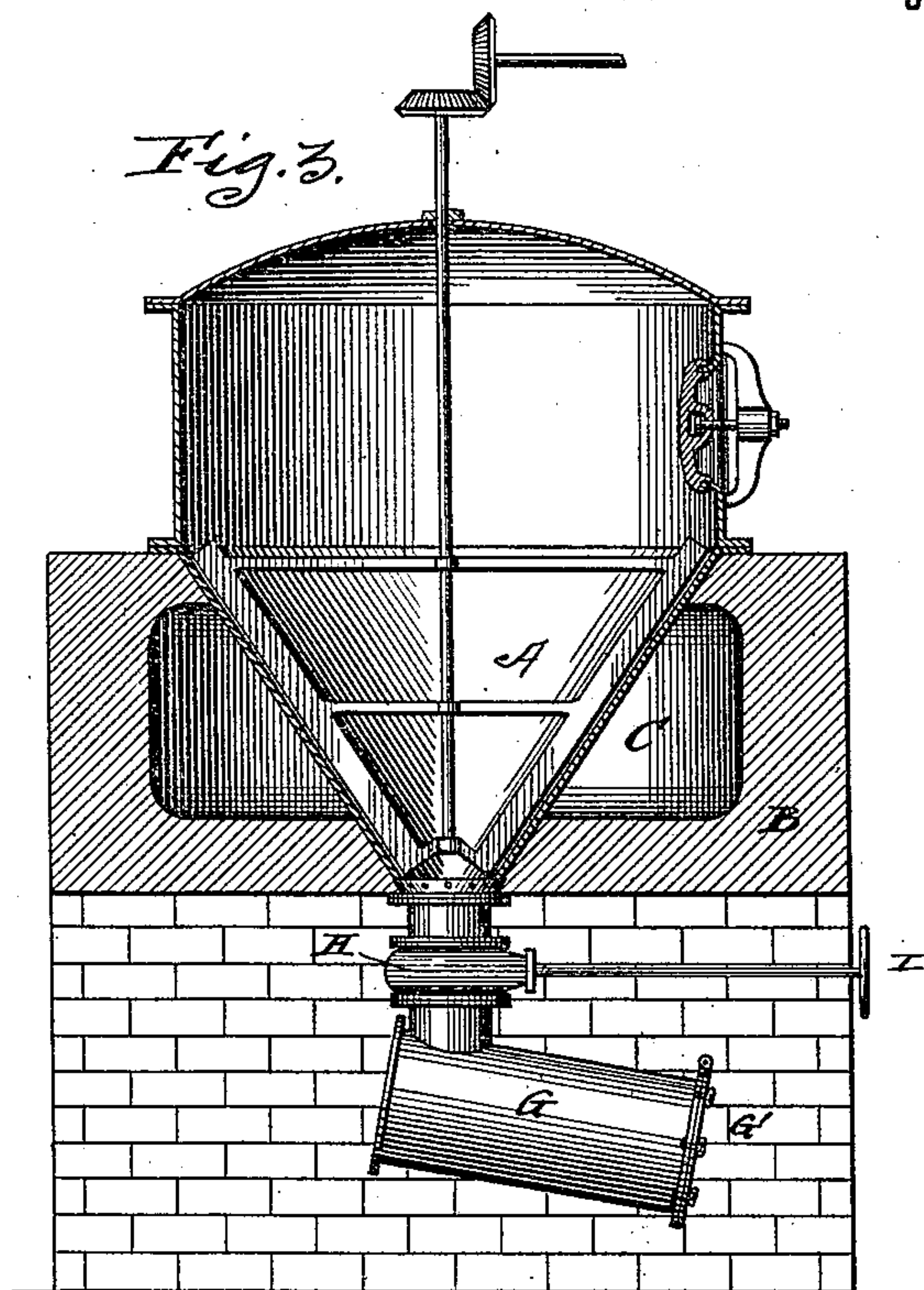
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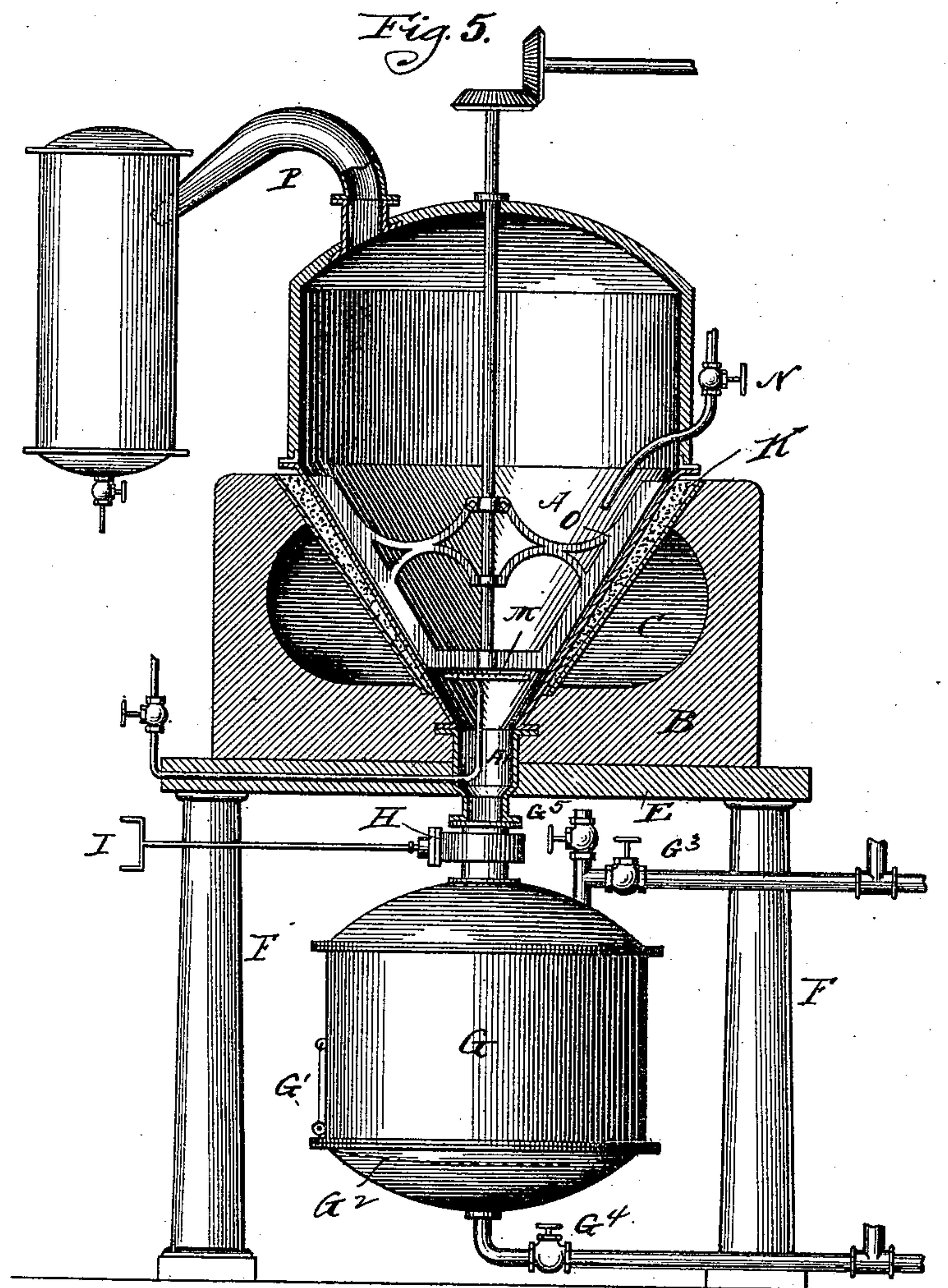
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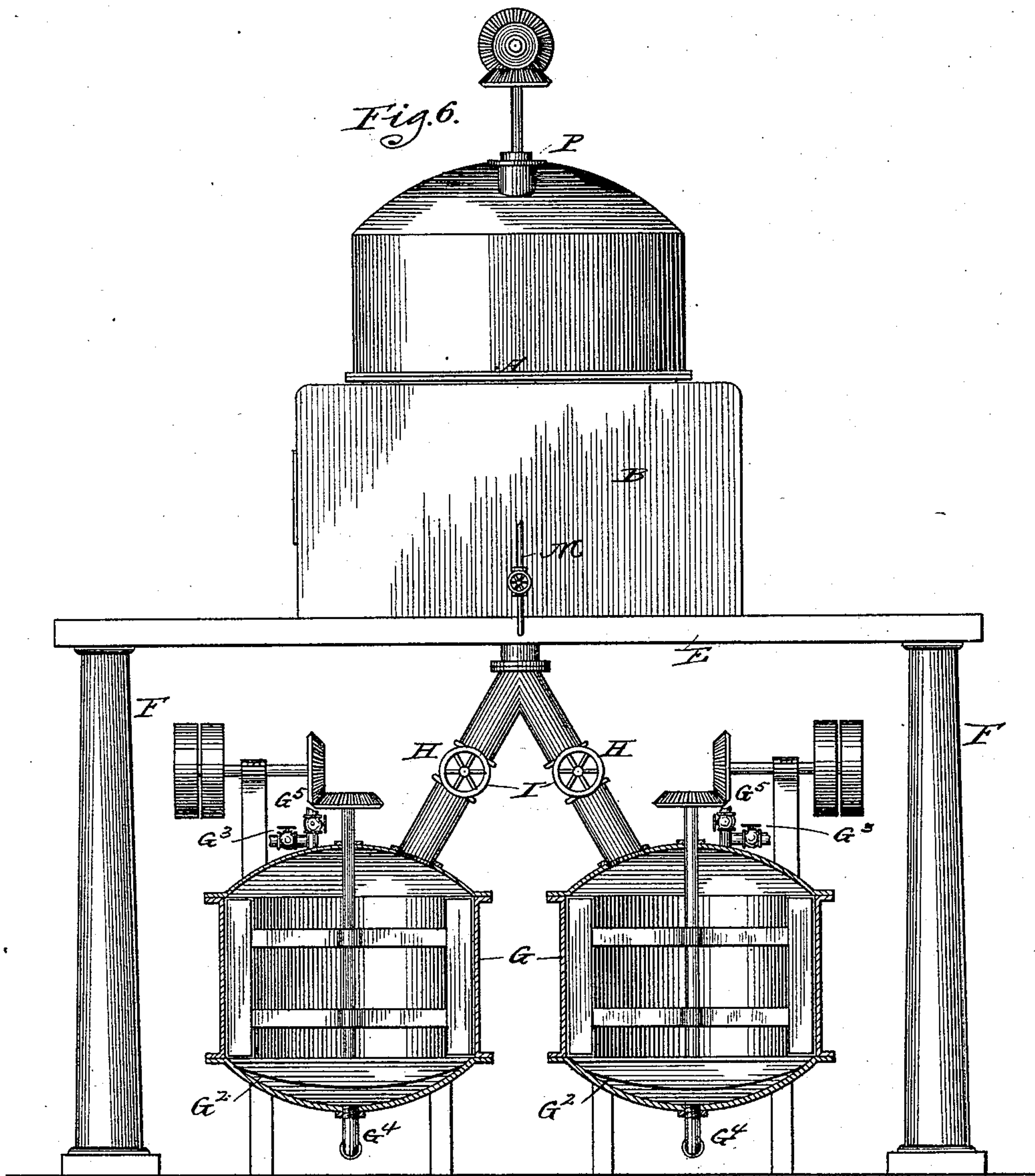
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UNITED STATES PATENT OFFICE.

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APPARATUS FOR THE DISTILLATION OF CONCENTRATED SOAP-LYE.

SPECIFICATION forming part of Letters Patent No. 428,470, dated May 20, 1890.

Application filed September 9, 1889. Serial No. 323,373. (No model.)

To all whom it may concern:

Be it known that we, ALBERT DOMEIER, merchant, and OTTO CHRISTIAN HAGEMANN, chemical engineer, both of London, England, have invented certain new and useful Improvements in Apparatus for the Distillation of Concentrated Soap-Lye or the Glycerine Recovered from Soap-Lye, of which the following is a specification.

10 The object of this invention is to provide an improved apparatus for the distillation of concentrated soap-lye or the glycerine recovered from soap-lye.

15 Concentrated spent soap-lye, or the crude glycerine recovered therefrom, contains a considerable quantity of mineral matters or salts—*e. g.*, chloride of sodium or sulphate of sodium—which it holds in solution. Under the influence of heat in the process of distilling the glycerine is volatilized and the salts are precipitated, and this precipitation of salt forms a serious obstacle in the way of successful working. The salts being allowed to accumulate in the vessel wherein the distillation is carried on, they cake together, and the cakes or scales inclose much glycerine, which is thus lost, and if the accumulation takes place in the vicinity of heated surfaces or superheated steam the glycerine is readily damaged by such portion as may be inclosed in the cakes or scales or in contact therewith getting too large a share of heat, and thus becoming burned or decomposed. Even with the best-known apparatus arranged for drawing off such accumulations of salts at intervals from the distilling-vessel by means of a valve or similar device such decomposition cannot be entirely avoided, and, besides, such apparatus has the additional disadvantage that a comparatively large quantity of the liquid contents of the distilling-vessel must be unavoidably drawn off together with the salts, and this has to be separated from said salts and returned to the still, these operations entailing extra expense and loss of time and heat. Moreover, the employment of a valve or similar device to periodically remove the precipitated salts from the distilling-vessel renders it impossible to work such an apparatus under a vacuum and very difficult under pressure, while, as is well known,

a vacuum is a desirable condition to be established in the distillation of glycerine, and we have found pressure also very useful with some special qualities of raw material. Again, in attempting to run such an apparatus continuously by feeding in charges of fresh liquor to take the place of the accumulated salts previously withdrawn the contents of the still are apt to froth up and boil over, owing to the freshly-introduced glycerine suddenly parting with its water under the influence of the increased temperature to which it is suddenly exposed, and thus loss and inconvenience are caused. Consequently, to avoid as far as possible these drawbacks, the operation has to frequently be stopped and the apparatus cleaned from the objectionable accumulations of salts, and such stoppages cause loss of time and heat and diminish the working capacity of the distilling-vessel.

The object of this invention is to overcome all these difficulties and to provide an apparatus to distill the concentrated soap-lye or glycerine in a more perfect manner than has hitherto been accomplished. This we effect by dividing our apparatus into two portions or vessels connected or separable at will by a valve or similar device, and apply heat to and effect the distillation in the upper of these two vessels only, while the lower chamber or vessel is out of the influence of the heat and forms a receptacle for the precipitated mineral matters or salts, which fall at once into it by virtue of their superior specific gravity, and thus do not remain in contact with the heated surfaces or superheated steam in the distilling portion of the apparatus. When the lower chamber or vessel is filled or nearly filled with such salts, we close the connection with the distilling-vessel and remove the salts, which are in a crystalline condition as precipitated, and all caking together and burning of such salts and consequently decomposition of glycerine from this cause have been entirely avoided. We have also found it advantageous to make the lower chamber or vessel in duplicate, so that one such chamber can be in open connection with the distilling chamber or vessel while the valve connecting the other one is closed for

the purpose of removing the salts or otherwise, and thus the salts can be more leisurely dealt with, and in one form of our apparatus we provide means in the lower chamber or vessel for washing such salts before their removal therefrom. After removing a charge of the precipitated salts from the lower chamber or vessel it may be filled with a fresh charge of the liquor to be distilled and the valve connecting it with the distilling-chamber again opened, thus introducing a fresh charge of liquor into the apparatus without appreciable loss of heat or disturbing the surface-line of the distilling portion or in any way interrupting the process, which goes on continuously, the liquor in the upper or distilling vessel or chamber being maintained in a state of ebullition at the distilling temperature and that in the lower chamber remaining quiescent and being gradually displaced and forced into the distilling-chamber by the precipitated salts which fall therefrom, and all frothing or boiling over and lowering of temperature which would be attendant upon introducing a large charge of fresh liquor directly into the distilling chamber or vessel is entirely avoided.

We will now describe our apparatus.

In the accompanying drawings, Figures 1 and 2 are respectively a vertical section and plan of one form of the apparatus fitted with one lower chamber or vessel to hold the quiescent portion of the liquor and to receive the precipitated salts, which are removed to a separate apparatus for washing. Figs. 3 and 4 represent apparatus with two such lower chambers or vessels for the quiescent portion of the liquor and receiving the precipitates. Fig. 5 represents a vertical section of an apparatus with the lower chamber or vessel arranged for washing the precipitated salts previous to their removal, and Fig. 6 represents a front view (partly in broken section) of a similar apparatus fitted with two lower chambers or vessels and with mechanical agitators for the more perfect mixing of the washing-liquors with the salts.

A is the still or vessel in which the distillation is effected, B the inclosing brick-work, and C the flue or heating space.

E and F, Fig 5, are respectively girders and columns to carry the apparatus when that form of construction is desired; otherwise the whole may be carried on brick-work.

G is the lower chamber or vessel, which contains the quiescent portion of the liquor under operation, and into which the mineral matters or salts fall when precipitated from the upper portion, and which can be separated from the upper portion by closing the valve H by means of the hand-wheel I.

G' is a door through which the collected mineral matters or salts in G can be removed, and G² a strainer or perforated plate allowing the passage of liquor to the bottom of the vessel, but retaining the mineral matters or salts.

G³ is a pipe through which brine or purified spent soap-lye containing less glycerine than the liquor adhering to the salts, or pure brine, may be admitted for the purpose of washing the crystals of mineral deposits or salts free from the glycerine-liquor adhering to them, the salts remaining upon and above the perforated plate or filter G² and the liquor falling through the filter and escaping by the pipe G⁴, from which it may be collected for reuse in any convenient manner.

G⁵ is a cock for allowing the ingress or egress of air to or from G.

K is a sand bath surrounding the distilling-vessel A, and is preferably used for the purpose of modifying the action of the fire upon the heated portion of the vessel A, and M is a steam-pipe and perforated coil for the introduction of superheated steam, and N is a pipe for feeding in fresh glycerine-liquor directly into A.

O is a mechanical agitator or scraper, the action of which will be well understood without special description.

P is the exit through which the vapors of steam and glycerine pass to the condenser or condensers.

The shape of the said portions of the apparatus may vary, as also the material of which they are constructed and the mode of applying the heat to the distilling portion. For instance, where it is desirable not to employ direct fire in the same building where the apparatus is situated the part A may be jacketed and heated entirely with superheated steam, or under some circumstances the outside heat may be entirely dispensed with and the distillation effected by means of the injected superheated steam only; and as to materials of construction we have employed cast and wrought iron, steel, and copper; but other materials may be used, and the apparatus may be provided with thermometers or pyrometers for controlling the temperature, and gages for vacuum and pressure, as is well understood.

The operation is as follows: The vessels composing the apparatus being all charged with the concentrated soap-lye or glycerine and the valves H being open, heat is applied to the vessel A and superheated steam introduced through the pipe and coil M, and the liquor in A brought to ebullition and heated to distilling-point, when the glycerine-vapors rise together with the steam and pass over through the pipe P to a suitable condenser or condensers, the level of the liquor under operation being maintained by the introduction in a comparatively small stream of fresh liquor through pipe N. During this operation the mineral matters or salts contained in the liquor are precipitated or thrown out of solution, and these fall through the valve H into the vessel G, displaying an equivalent quantity of the liquor in G and forcing it upward into A. This action proceeds until G is filled or nearly filled with said precipitated

salts, when the valve H is closed, and in the case of using the apparatus as shown in Figs. 1, 2, 3, and 4 the door G' is opened and the salts withdrawn and removed to a suitable apparatus for washing them.

In the case of using the apparatus of Figs. 5 and 6 the valve H is closed and the valves G⁵ and G⁴ are opened, and the glycerine-liquor adhering to the salts is drawn off or allowed to run off through the pipe G⁴ to a suitable receptacle. G⁴ is then closed and a suitable quantity of washing-liquor run in through the pipe G³, which is then closed, and G⁴ is again opened and the washing-liquors withdrawn for reuse or concentration. This operation is repeated as often as may be necessary, and finally the door G' is opened and the washed salt removed. G is now filled with fresh glycerine-liquor through the pipe G⁴ or G³, or otherwise by a special pipe, and, the valves G³, G⁴, and G⁵ being closed, the valve H is opened, and distillation, which has not been interrupted during the removal of the salts, proceeds as usual. In the case of the use of the apparatus Figs. 3, 4, and 6, with two chambers G, these are used alternately, as will be readily understood, while in the forms of apparatus Figs. 1, 2, and 3 the pocket A' is provided for the reception of any small quantities of salt, which are precipitated during the time the valve H is closed.

The vessel or duplicate vessels G may be mounted with mechanical stirrers for mixing the salts with the washing-liquor, as shown at Fig. 6, or such mixture may be effected by blowing air through the mass, for instance, by way of the pipe G⁴, the air afterward escaping by the cock G⁵, and the removal of the washing-liquor may be effected by direct suction or by air or similar pressure from above, or, where time permits, by gravitation without pressure.

Instead of filling fresh concentrated lye or glycerine into the vessel G after each time of removal of the salts therefrom and to fill the space thus made vacant, the valve H may be opened, allowing a portion of the liquor in A to flow down and fill G, the air in G escaping upward through A and passing over into the condensers, or by preference it escapes through the cock G⁵, which is closed when the vessel G is filled. In this case the level of the liquor in A will be lowered, and may be corrected to the proper point by accelerating the flow through the pipe N when and as fast as the nature of the liquor permits without causing the charge to boil over, and to this end it is desirable to heat the liquor before it enters the vessel A to as near the distilling-point as possible; but this method of introducing fresh liquor to fill the space rendered vacant by the withdrawal of a charge of precipitated salt and apart from the regular inflow of the quantity constituting the regular feed to the vessel A is not recommended, for, although the hot liquor which flows down from A to fill G will in due course be forced upward again

into A by the precipitated salt, there is an inevitable loss of heat by radiation from G, and the disturbance of the level of the liquor in A is liable to cause variations in the quality of the distillate therefrom.

The apparatus may be worked at the normal or diminished or increased atmospheric pressure without interrupting or otherwise interfering with the continuity of the process.

We claim—

1. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination, with a still or vessel wherein the distillation is effected, of a lower closed vessel communicating therewith by a valve-controlled passage, said lower vessel having a discharge-door.

2. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination, with a still or vessel wherein the distillation is effected, of a lower closed vessel or vessels communicating therewith by a valve-controlled passage or passages, said lower vessel provided with induction and eduction pipes with suitable valves, and a door through which to remove the matters precipitated in the distillation, substantially as described.

3. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination, with a still or vessel wherein the distillation is effected, of a lower closed vessel communicating therewith by a valve-controlled passage, said lower vessel having a strainer, substantially as described.

4. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination, with a still, of a lower closed vessel communicating therewith by a valve-controlled passage, said lower vessel provided with an agitator, substantially as described.

5. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination of a still having side walls converging toward its lower end, a discharge-opening in said end, and a lower closed vessel communicating with the still by a valve-controlled passage, said lower vessel having a discharge-door, substantially as described.

6. In an apparatus for the distillation of concentrated spent soap-lye or glycerine, the combination of a still or vessel wherein the distillation is effected, said vessel having side walls converging toward its lower end and terminating in an extension forming a pocket, and a second closed vessel communicating with the still by a valve-controlled passage, substantially as described.

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