

(No Model.)

4 Sheets—Sheet 1.

E. A. BARBET.

PROCESS OF AND APPARATUS FOR RECTIFYING AND DISTILLING
ALCOHOL, &c.

No. 436,764.

Patented Sept. 16, 1890.

FIG. 2.

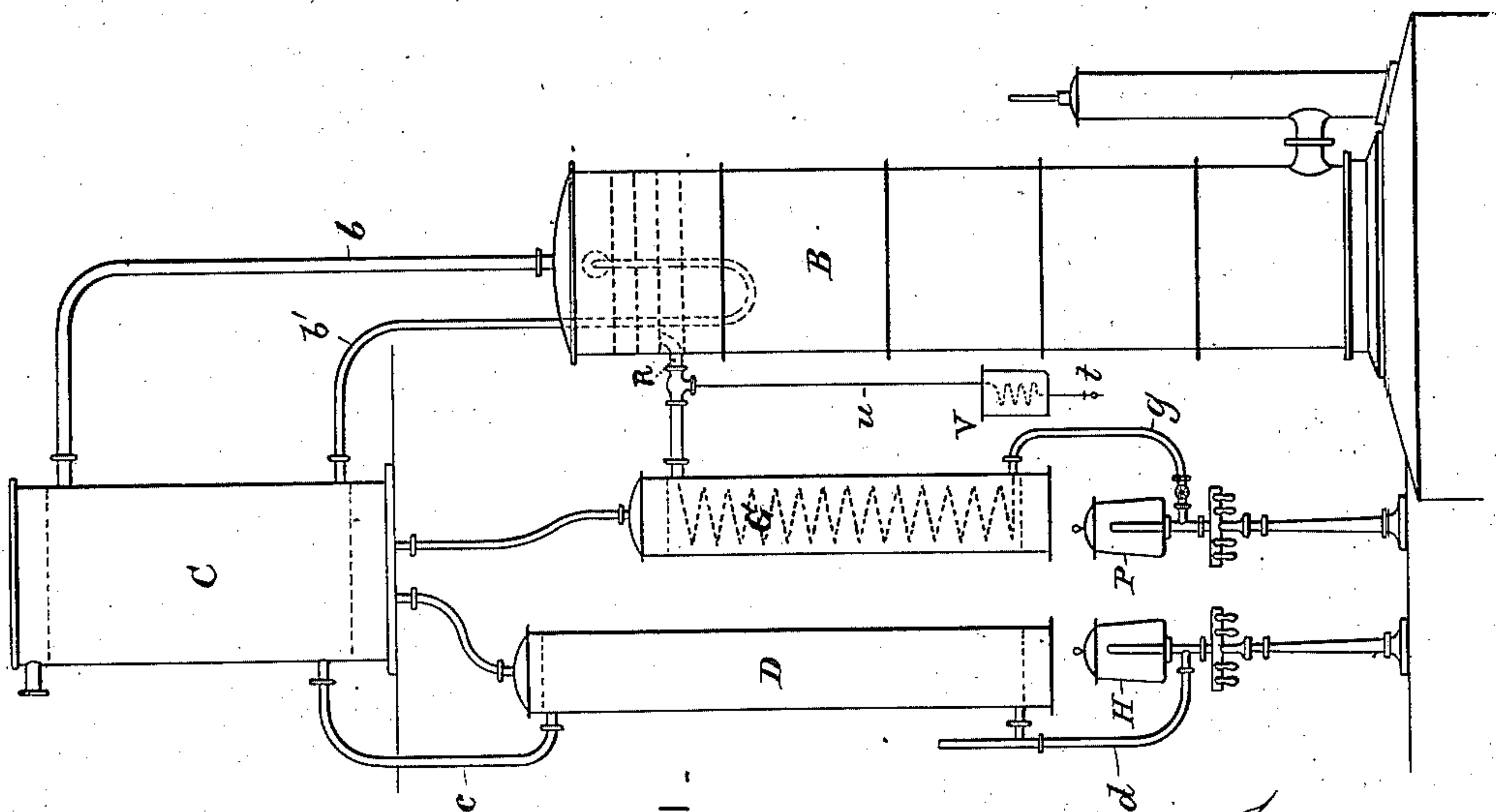
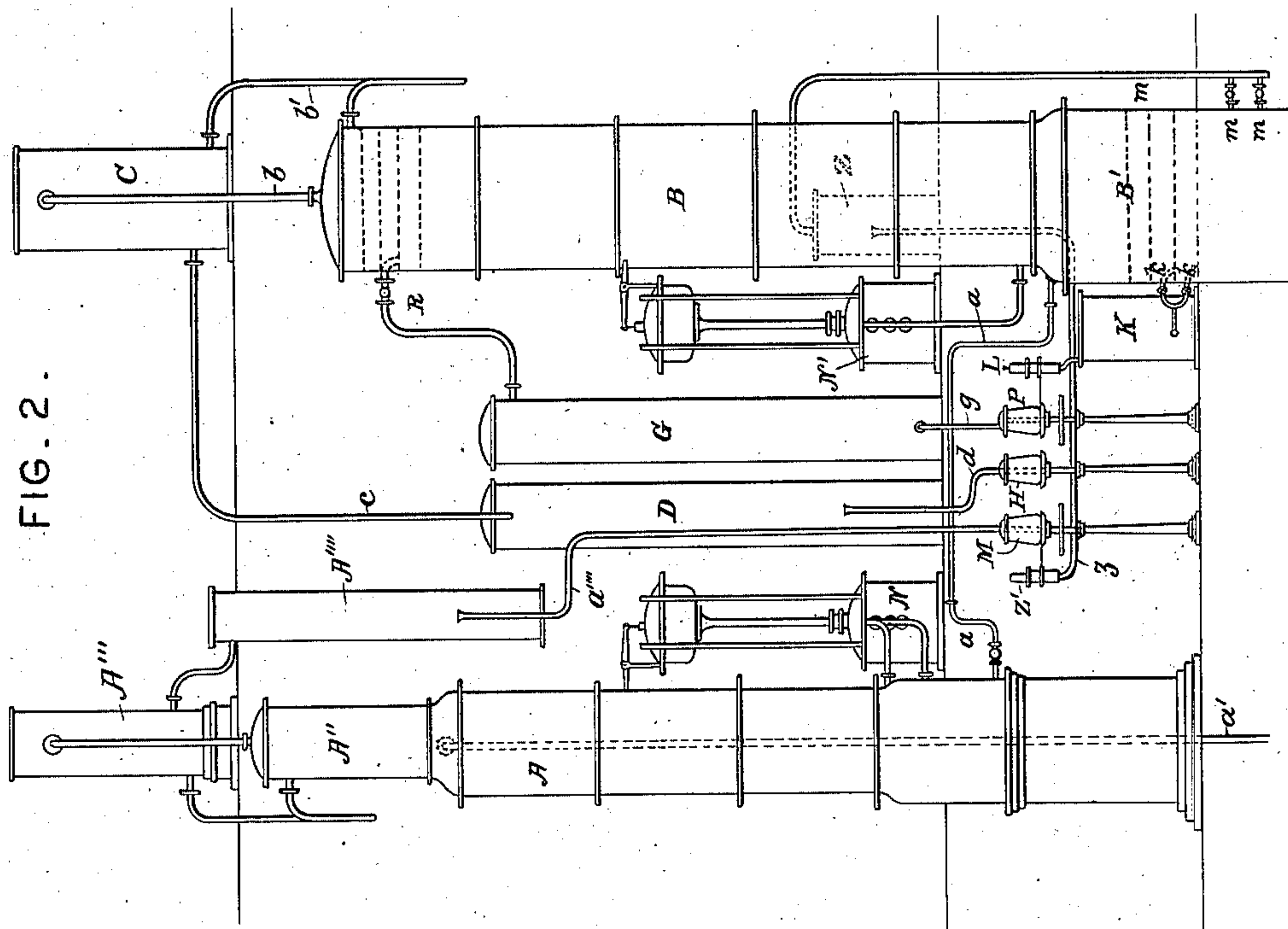


FIG. 1.

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by Pollock & Harris
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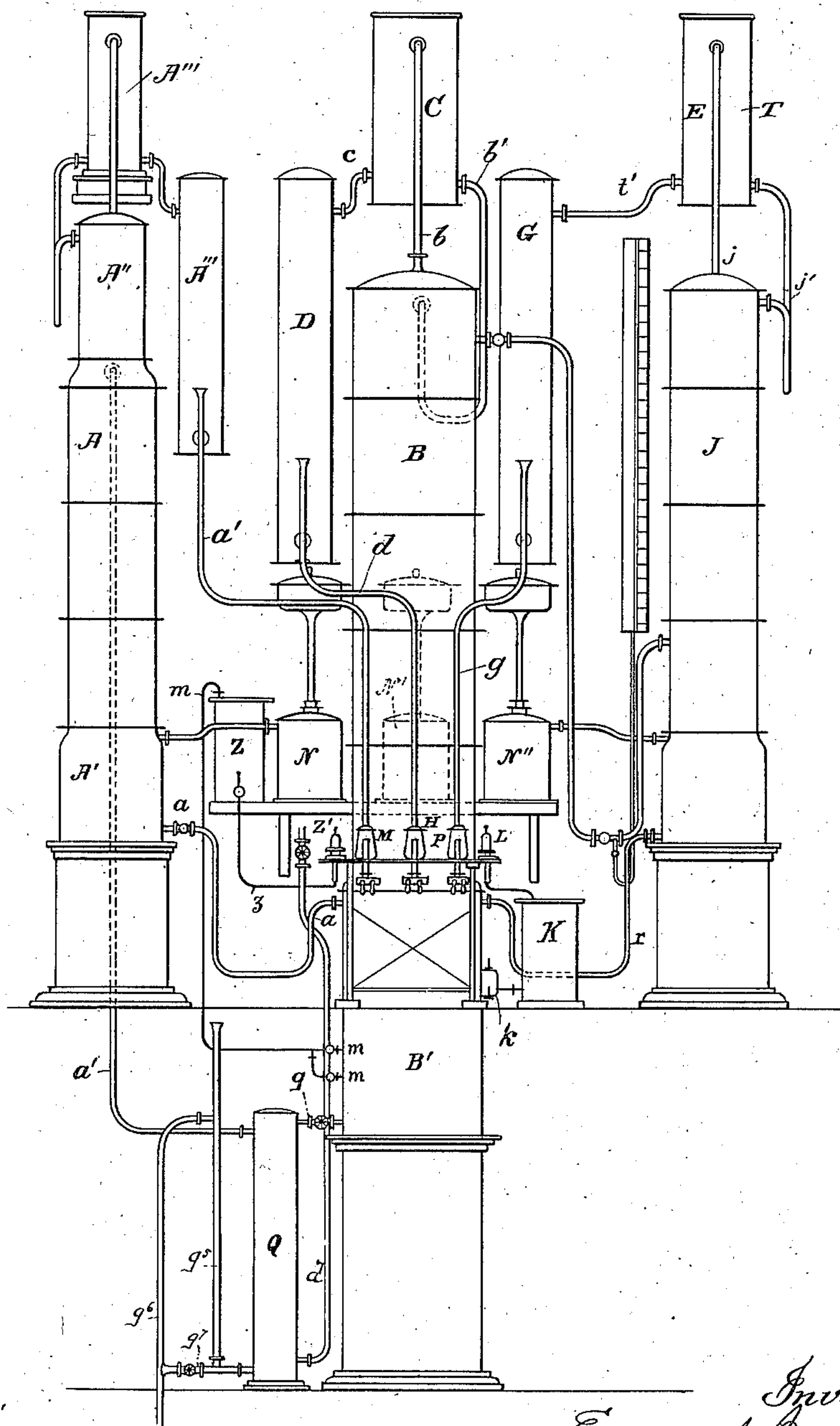
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FIG. 3.



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FIG. 6.

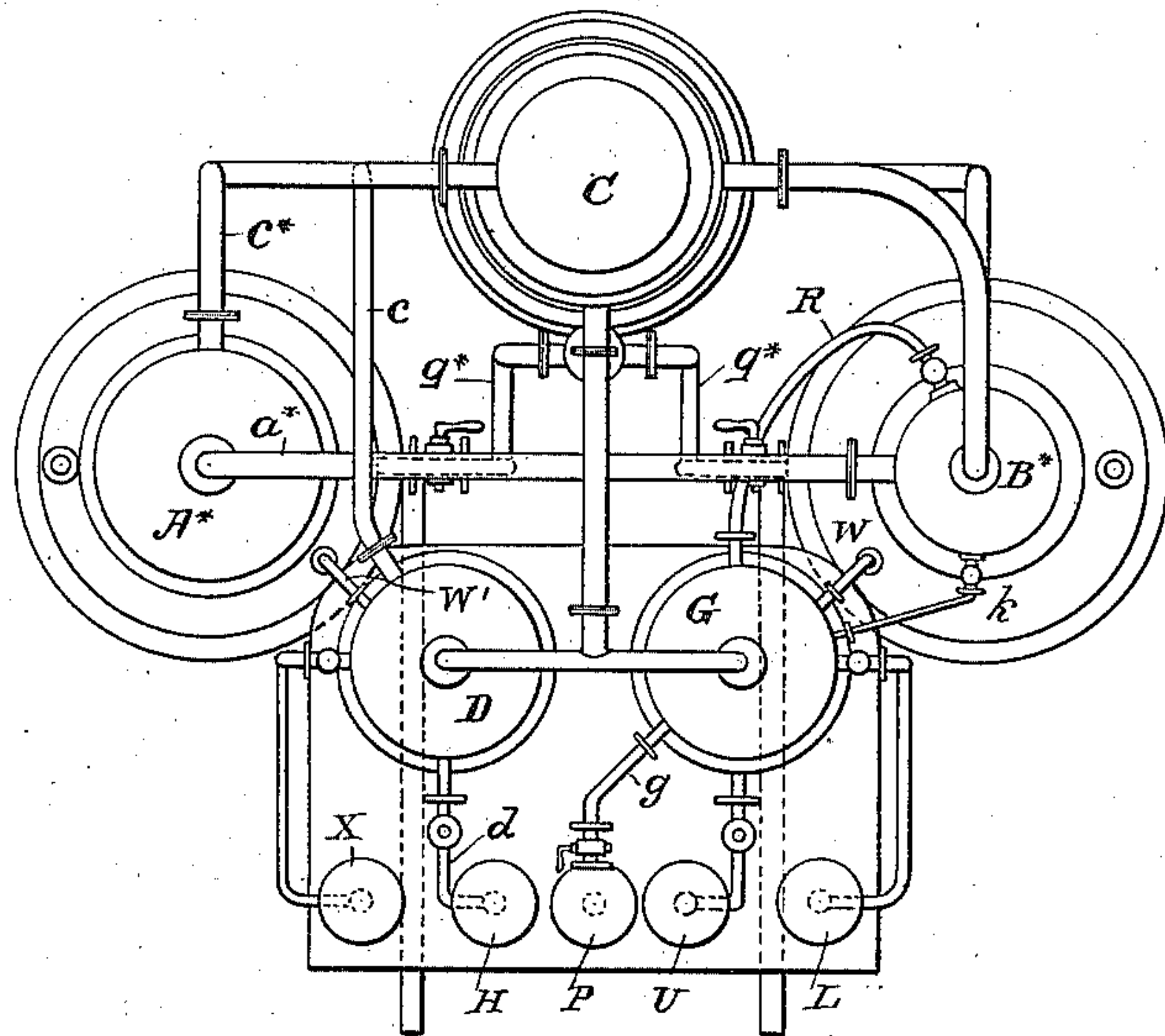
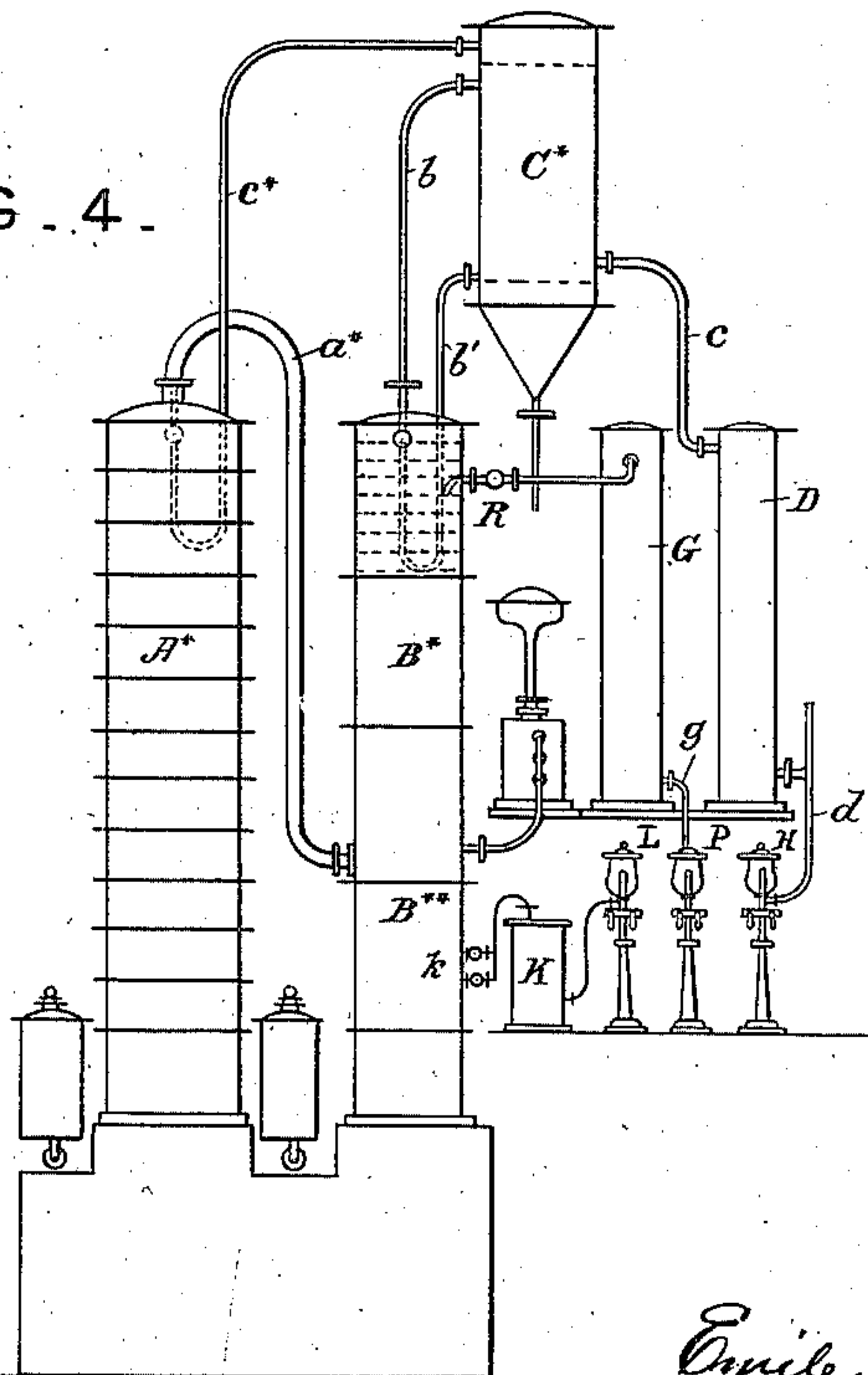


FIG. 4.



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4 Sheets—Sheet 4.

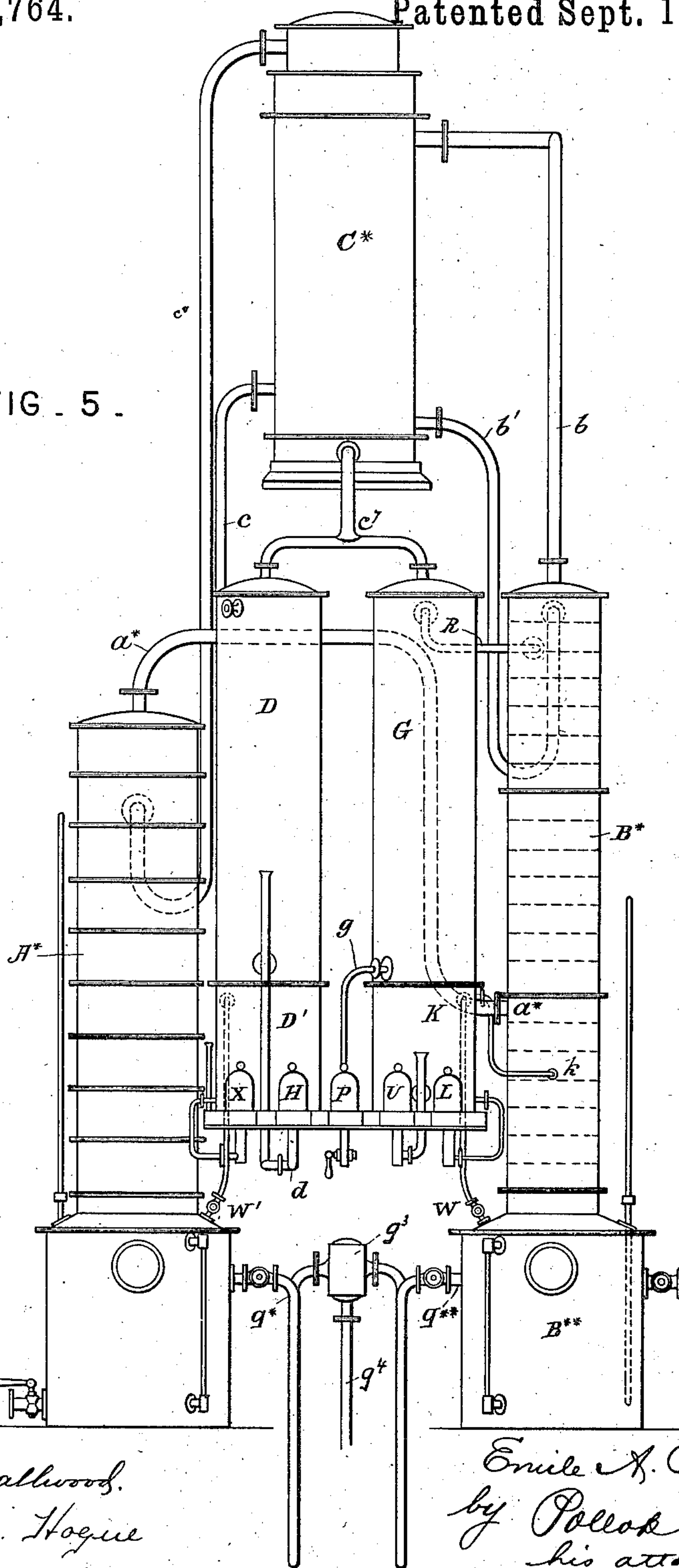
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FIG. 5.



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UNITED STATES PATENT OFFICE.

EMILE AUGUSTIN BARBET, OF PARIS, FRANCE.

PROCESS OF AND APPARATUS FOR RECTIFYING AND DISTILLING ALCOHOL, &c.

SPECIFICATION forming part of Letters Patent No. 436,764, dated September 16, 1890.

Application filed March 20, 1890. Serial No. 344,629. (No model.) Patented in France December 21, 1888, No. 195,113; in Belgium January 28, 1889, No. 84,793; in England March 15, 1889, No. 4,549; in Germany April 26, 1889, No. 52,632, and in Austria-Hungary April 11, 1890, No. 54,774 and No. 13,424.

To all whom it may concern:

Be it known that I, EMILE AUGUSTIN BARBET, a citizen of France, and a resident of Paris, in the Republic of France, have invented a new and useful Improvement in Process of and Apparatus for Rectifying and Distilling Alcohols and other Volatile Products, (for which I have obtained patents in France, No. 195,113, dated December 21, 1888; in Belgium, No. 84,793, dated January 28, 1889; in Great Britain, No. 4,549, dated March 15, 1889; in Germany, No. 52,632, dated April 26, 1889, and in Austria-Hungary, No. 54,774 and No. 13,424, dated April 11, 1890,) which improvement is fully set forth in the following specification.

This invention relates more particularly to the treatment of different kinds of spirits or brandies from fermented liquors of various kinds, and has reference both to distillation and to rectification. By means of said invention alcohols or spirits of very great purity may be obtained in rectification and the crude spirits or brandies from first distillations are considerably improved. Brandies from wine and from fruit, rums, liquors, &c., also may be obtained of great purity and fine flavor.

In the accompanying drawings, which form part of the specification, Figure 1 is an elevation of a rectifying apparatus constructed or modified in accordance with the present invention. Fig. 2 is a similar view of apparatus for continuous rectification. Fig. 3 is a similar view of a continuous rectifying apparatus provided with additional improvements. Fig. 4 is a similar view of a distilling apparatus. Figs. 5 and 6 are an elevation and a plan, respectively, of a distilling apparatus with additional improvements.

Referring to Fig. 1, the column B of a rectifier is connected in the ordinary way by the vapor-escape pipe *b* and liquid-return pipe *b'* with the partial condenser or dephlegmator C, and this by the pipe *c* with the refrigerator or condenser D, which in turn communicates by the pipe *d* with the proof-bottle H.

I have discovered that if the alcoholic liquid from the first plates at the top of the rectifying-column be drawn off—say by the outlet-pipe R (additional to the vapor-escape *b*)—

this liquid is very much more free from the light products than the alcohol which runs off by way of the proof-bottle H. It suffices to cool this alcohol by an appropriate cooler G, and collect it through the pipe *g* and proof-bottle P, in order to have a better alcohol than that from the proof-bottle H.

At Y is a small cooler connected by the pipe *y* with the pipe R, and terminating in a small test-cock *t*.

The difference between the liquid of retrogradation drawn from the upper plates of the rectifier and that which would be obtained from the proof-bottle H if the outlet-pipe R were closed is analogous to the results of purifying spirits by aging, and is comparable with the effect of aging wines by the process of M. Pasteur. This process of extraction or drawing off at a suitable point below where the return-pipe *b'* enters the column so as to receive the returned liquid can be applied to an ordinary discontinuous rectifier; but this demands a special oversight on the part of the operator, because of the perpetually-changing conditions which attend this sort of rectification. In particular it is to be noted that as the amylic oils at the end of each rectification come over it is necessary to stop this sort of extraction for a time. Besides, at the commencement of a rectification, since the plates are left foul by the after-runings of the preceding operation, it is necessary to wait still for some time until the bad odors are carried off by the alcoholic vapors themselves. For continuous rectification, however, the withdrawal of the liquid of retrogradation presents greater advantages, since the yield is constant and there is no special oversight to be maintained, and the alcohol so withdrawn is of very great purity with respect to light products.

Referring to Fig. 2, the crude spirits are deprived of the products more volatile than alcohol in the column-still A, provided with a feed-pipe *a'*, a small rectifying-column A'', a partial condenser or dephlegmator A''', a condenser or cooler A''', and a pipe *a''''*, leading therefrom to a proof-bottle M. At N is shown the regulator (of ordinary construction) for governing the supply of steam to the

still. The crude spirits, deprived of their light impurities in the still A, are fed to the rectifier B by the feed-inlet pipe a , which is also the outlet-pipe for the still A. The parts C, D, H, R, G, and P are the same as in Fig. 1, and in general parts similarly lettered are similar in all the figures.

Below the feed-inlet a of the rectifying-column B and communicating with the lower part of the said column is a still or vaporizer in the form of a steam-heated column B', having a spent-wash outlet for the exhausted liquid, (such as shown at q in Fig. 3, the bottom of the column being omitted in Fig. 2,) and provided at suitable points, as at k and m , above said outlet with draw-offs. The draw-off k is for conducting the amylic oils in liquid form to the cooler K, whence they pass to the proof-bottle L. By the draw-off m vapor may be conducted into the condenser or cooler Z, (which communicates with a proof-bottle Z' by pipe z), in order to ascertain the degree of exhaustion of the spent liquid.

At N' is the regulator for governing the admission of steam to the distilling or vaporizing column B'. The column B is supplied at the feed-inlet a with a stream of partly-purified crude spirits, the volatile constituents of which are vaporized, and the vapors, after being rectified in their passage upward by the successive partial condensations which eliminate the heavy impurities, are partly condensed in the apparatus C, whereby the alcoholic portion is separated from the lighter impurities, and this condensed liquid, being returned to the top of the rectifier B, is drawn off from the upper plates thereof by the outlet-pipe R. The traces of lighter impurities which may have remained in the spirits from the still A find their escape by way of the proof-bottle H. The heavier impurities eliminated in the column B are carried down and drawn off below.

The alcohol of retrogradation which escapes by the proof-bottle is equivalent in freedom from lighter impurities to alcohol of double rectification; but with respect to the heavier impurities it has been subjected only to a single rectification, and consequently is not chemically pure. To bring it to absolute purity with reference to the heavier as well as the lighter impurities, it suffices (see Fig. 3) to interpose a rectifier J between the outlet R for the pasteurized alcohol from the rectifier B and the cooler G and proof-bottle P. This rectifier, as shown, is provided with a still or vaporizer in the form of a steam-heated column J communicating with the lower part of the column J, and also with a condenser or dephlegmator T connected in the usual way with the upper part of said column J by the vapor-escape j and the return-pipe j' and by the pipe t' with the condenser or cooler G.

At N'' is the regulator for governing the admission of steam for heating the column J'. The alcohol withdrawn from column B by the

outlet R, in place of being sent directly into the cooler G, is subjected to a supplementary rectification in the column J, the alcoholic vapors ascending through the plates, and the heavier impurities which have been retained being eliminated by the successive partial condensations. The purified alcohol passes through the condenser or cooler G to the proof-bottle P. The heavier impurities descend to the base of the rectifier, and to prevent their accumulation therein a return-pipe r discharges them into the rectifier B. A small cock suffices to regulate the return-flow through this pipe, as the volume is very small. At Q is a heat exchanger or regulator for heating the liquid to be supplied to the column A by means of the spent liquid from the rectifier B. The cooled spent wash passes away by the pipes q^5 q^6 , which keep the regenerator Q full of spent wash. At q^7 is a direct connection provided with a valve which is ordinarily closed, but which may be opened whenever it is desired to draw off the spent liquor from the said regenerator. Liquid from column A enters the regenerator by the pipe a^7 and passes to the column through the pipe a' .

In consequence of the removal of the lighter impurities, and the complementary rectification in the column J for the heavier impurities, the alcohol obtained at the proof-bottle P is, in the full sense of the word, alcohol of double rectification chemically pure and neutral, and this result is obtained very economically.

The processes described are applicable by extension to distilling-columns for industrial alcohols or for fruit (including grape) brandies and other liquors.

In column apparatus which yield strong crude spirits it suffices to apply thereto the purification by means of the outlet R, cooler G, and proof-bottle P, and the extraction of the heavy oils by means of the pipes k , cooler K, and proof-bottle Z. In Fig. 4 the distilling-column A* is supplied by the pipe c^* from the wash-heater C*, which also acts as a partial condenser or dephlegmator for the rectifying-column B*. The vapors from the still A* pass through the pipe a^* into the rectifying-column B*, and having been purified and concentrated therein by elimination of the heavier impurities pass into the wash-heating dephlegmator C*, by means of pipe b'' , from which the condensed alcoholic liquid is returned to the column B* by pipe b' , while the lighter impurities pass through the condenser or cooler D to the proof-bottle H. The alcohol which passes by the pipe R and cooler G to the proof-bottle P will be a fair grade of rectified alcohol if the fermented material is of good quality. Communicating with the lower part of the column B* is a still or vaporizer in the form of a steam-heated column B**, provided with the draw-off k for the amylic oils.

For the special purification of wine, brandy, and tafia the arrangement of Fig. 5 is adopted.

All wines are not, or have not, been capable

of yielding cognac brandy, and particularly is this true of the wines of the Midi, which are generally fermented at too high a temperature. In them are found impurities higher than alcohol and also amylic impurities, like the products from the industrial fermentation of grains, beet-roots, or molasses. By the new or improved process of withdrawing the alcohol of retrogradation and extraction of the oils it is possible to remove the bad odors, to age the brandy, and to deprive it of the taste of the lees, while at the same time preserving the bouquets and aromas desired by consumers. By like means it is possible to obtain from the fermented liquor, or liquor from sugar-cane molasses or from sugar-cane itself, a true rum, aged and free from all bad odors.

Referring to Figs. 5 and 6, the fermented liquor from the wash-heating dephlegmator C* is introduced through the pipe c^* into the column-still A*, and is therein exhausted of its alcohol, the vapors passing by the pipe a^* to the rectifier B*. The alcoholic vapors, concentrated and purified more or less from the heavier impurities by the action of the rectifying-plates, pass into the partial condenser or dephlegmator C*, from which the condensed liquid is returned to the rectifier, while the lighter products pass to the condenser or cooler D, and thence to the proof-bottle H. As shown, a branched pipe c^7 connects the tops of coolers D and E with the lower part of the dephlegmator C*, for conducting into said dephlegmator the liquid which has been already utilized to reduce the temperature in the coolers D and G. By the pipe R the purified alcohol is withdrawn. It is cooled in the vessel G and passes to the proof-bottle P.

At a lower point on the still or vaporizer B** the fetid oils (amylic, butylic, and essence of the lees) are withdrawn by the pipe k , which conducts them to the cooler K. From the cooler K they pass to the proof-bottle L. Still lower down the heavier ethers—œnanthic, pelargonic, raisin essence, &c.—are collected, they being taken off by the pipe W and conducted to a coil in the cooler K alongside of that for the amylic oils. On the large chamber at the base of the column A* a vapor-collector or draw-off W' is also provided, which has for its object not only the verification of the proper exhaustion of the liquor, but also the drawing off of the heaviest ethers and essences which are particularly hard to distil. They pass through the condenser or cooler D' to the proof-bottle X. These essences of slight volatility from the two stills are agreeable to the taste, and characteristic of cognac brandy. These liquids from the two proof-bottles X and U are added to the alcohol from the proof-bottle P, they seeming to dilute the spirit and increase its specific gravity and to restore to it the pleasant bouquets and aromas or essences. The brandy or rum thus obtained is free from both the lighter impurities and the amylic impurities while having the agreeable perfumes characteristic of these liquors. Thus

an important industrial result is attained by the process. At the base of each of the columns is a pipe q^* q^{**} , respectively, for drawing off the spent liquor. They are siphon-pipes and empty into an intermediate chamber q^3 , from which a discharge-pipe q^4 leads away.

In place of connecting the column with outlet R directly with the column for exhausting the liquid to be distilled or rectified, it is clear that two separate or indirectly connected apparatus may be used. In this case the first column would be operated to make weak crude spirits, which would be cooled, delivered by a pump into an elevated vat and thence fed to a column provided with a dephlegmator and an outlet for the alcohol of retrogradation, as shown, for the columns B* or B'', exactly as it would be fed to the purifiers or continuous rectifiers. A heat-exchanger or regenerator may also be used as a preliminary heater for the crude spirits. This arrangement is to be preferred in many cases—notably, first, in works having a column producing weak spirits, for the rectifying apparatus with means for withdrawing the liquid of retrogradation can then be set up without interfering in any way with the distillatory column; second, in countries where the industrial arts are but little cultivated, for it is much easier to attend to two independent or indirectly connected apparatus than where they are directly connected. In this latter case it is in fact necessary to have intelligent and attentive workmen for distillers in order to get good results. Further, the rectifying-column provided with means for withdrawing the liquid of retrogradation may be substituted for the purifier or column apparatus for removing the lighter impurities in the continuous rectifying process. In fact, experience shows that the expulsion of the light ethers is often better by such rectifying-column than by the purifier itself; besides, such column has the advantage over the purifier of getting rid not only of the lighter but of the heavier impurities as well. This double purification demands, it is true, a larger expenditure of steam, but there exist cases in which the crude spirits are so foul with the heavier impurities that this preliminary elimination of the fusel is most useful for improving the alcohol. It is easy to understand this arrangement, it being found in Fig. 3 by suppression of the column A and feeding the crude spirits directly to the rectifier B after the same have been heated in the regenerator Q.

Between the column B and the final column J a meter may be interposed to facilitate the conduct of the apparatus.

It is also possible to introduce free steam into the vaporizer or lower part of the rectifying-column instead of heating the same by a steam-coil. A permanent supplementary means for controlling the working of the apparatus may be secured by placing in the neighborhood of the feed-plates a thermom-

eter or thalpotasimeter. If the apparatus tends to become exhausted the temperature rises. If the feed is excessive, the alcoholic strength increases and the temperature of ebullition correspondingly falls.

I claim as my invention or discovery—

1. The process of carrying on the distillation of fermented liquids or rectification of crudespirits, consisting in vaporizing the volatile constituents, eliminating the heavier impurities from the mixed vapors by successive partial condensations in a rectifying-column, which heavier impurities are carried down the column and withdrawn below, separating the alcoholic portion from the lighter impurities by partial condensation in a condenser or dephlegmator returning the alcoholic portion to the top of the column for action of the ascending vapors, from which the heavier impurities have been removed, and withdrawing the alcohol from near the top of the column to avoid contamination by such heavier impurities, substantially as described.

2. The process of carrying on distillation or rectification, consisting in distilling off the lighter impurities from the fermented liquor or the crude spirits, evaporating the volatile constituents of the thus partially purified liquor or spirits, eliminating the heavier impurities by successive partial condensations in a rectifying-column, which heavier impurities are carried down and withdrawn below as a liquid, separating the alcoholic portion by partial condensation in a condenser or dephlegmator from the lighter impurities which have escaped the first operation aforesaid, returning the alcoholic portion to the action for a brief time of the ascending mixed vapors freed from the heavier impurities, and withdrawing the alcoholic portion near the top of the column, substantially as described.

3. The process of carrying on distillation or rectification, consisting in vaporizing the volatile constituents of the fermented liquid or crude spirits, eliminating the heavier impurities by successive partial condensations in a rectifying-column, which heavier impurities are carried down and withdrawn below, separating the alcoholic portion from the lighter impurities of the vapors by partial condensation in a condenser or dephlegmator, returning the alcoholic portion so separated to the top of said column, withdrawing it near the top and subjecting the same to a supplementary rectification, substantially as described.

4. The process of distillation or rectification, consisting in the following steps: vaporizing the volatile constituents, separating the alcoholic portion and lighter constituents from the heavier impurities, withdrawing the alcoholic portion from the mixed vapors by partial condensation, separating the amylic oils from the said heavier impurities, and finally distilling from the residuum the ethers

and essences heavier than amylic oils, and adding such products to the alcoholic portion, substantially as described.

5. The combination of a rectifying-column, a dephlegmator or partial condenser, a vapor-pipe connecting the two, a return-pipe for the condensed alcohol leading to the top of the column, and another pipe a short distance below the top of the column for withdrawing the alcohol, substantially as described.

6. A rectifying-column provided with an outlet from the upper part in addition to the vapor-escape, in combination with a dephlegmator or partial condenser connected with the said column through said vapor-escape, and having a return-pipe connected with the upper part of said rectifying-column, and a cooler connected with said rectifying-column through such outlet, substantially as described.

7. A rectifying-column, provided with an outlet from the upper part in addition to the vapor-escape and with a still or vaporizer communicating with the lower part of said column, in combination with a dephlegmator or partial condenser connected with the upper part of said column through said vapor-escape and also by a return-pipe, and a preliminary still or still and rectifier from which the liquid exhausted of certain constituents is delivered to said first-mentioned still or vaporizer, substantially as described.

8. A rectifying-column provided with an outlet from the upper part in addition to the vapor-escape, and with a still or vaporizer communicating with the lower part of said column, in combination with a dephlegmator or partial condenser connected with the upper part of said column through said vapor-escape and also by a return-pipe, and a preliminary still and rectifier from which the liquid exhausted of certain constituents is delivered to said first-mentioned still or vaporizer, substantially as described.

9. A rectifying-column provided with an outlet from the upper part in addition to the vapor-escape, in combination with a partial condenser or dephlegmator connected with the upper part of said column through said vapor-escape and also by a return-pipe, a supplementary rectifying-column connected with the first-named column through said outlet, and a cooler or condenser connected with the upper part of said supplementary rectifying-column, substantially as described.

10. A distilling or rectifying apparatus, comprising a rectifying-column provided with a feed-inlet at or near the base of said column and a still or vaporizer below said feed-inlet, also with a draw-off intermediate the said feed-inlet and said still or vaporizer, also with a vapor-escape, and an additional outlet near the top of the rectifying-column, and also with a dephlegmator connected with the said column through the said vapor-escape

cape and through a return-pipe entering the said column above said additional outlet, substantially as described.

11. A distilling or rectifying apparatus, comprising a rectifying-column provided with a feed-inlet at or near the base of said column and a still or vaporizer below said feed-inlet, also with two or more draw-offs at different points intermediate the said inlet and the said still or vaporizer, also with a vapor-escape and additional outlet near the top of the rectifying-column, and also with a dephlegmator or partial condenser connected with said column through said vapor-escape and through a return-pipe entering the said column above said additional outlet, substantially as described.

12. A distilling or rectifying apparatus, comprising a rectifying-column provided at its upper part with a vapor-escape and an additional outlet, in connection with a still or vaporizer communicating with the lower part of said column and provided with two draw-offs at least, in addition to and at different points above the spent-wash outlet, and a dephlegmator or partial condenser connected with the upper part of said column through said vapor-escape and also by a return-pipe, substantially as described.

13. A distilling or rectifying apparatus, comprising a rectifying-column provided with a vapor-escape and additional outlet at or near the top of said column, a dephlegmator or partial condenser connected with the

said column through said vapor-escape, and through a return-pipe entering the said column above said additional outlet, and two condensers or coolers and proof-bottles connected with said rectifying-column, one through the said dephlegmator or partial condenser and vapor-escape and the other through the said additional outlet, substantially as described.

14. A distilling or rectifying apparatus, comprising a column provided with a vapor-escape and an additional outlet at or near the top of said column, a dephlegmator or partial condenser connected with the said column through said vapor-escape and through a return-pipe entering said column at its upper part, a still or vaporizer communicating with the lower part of said column and one or more draw-offs above the spent-wash outlet of the said still or vaporizer, and three or more condensers or coolers and proof-bottles connected with said rectifying-column and still or vaporizer, one through the dephlegmator and vapor-escape, a second through said additional outlet, and the remainder through the said draw-offs, substantially as described.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

EMILE AUGUSTIN BARBET.

Witnesses:

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GEORGE R. OSCHERMER.