(No Model.) 4 Sheets—Sheet 1.

H. J. KREBS.

DISTILLING APPARATUS.

No. 589,000. Patented Aug. 31, 1897.

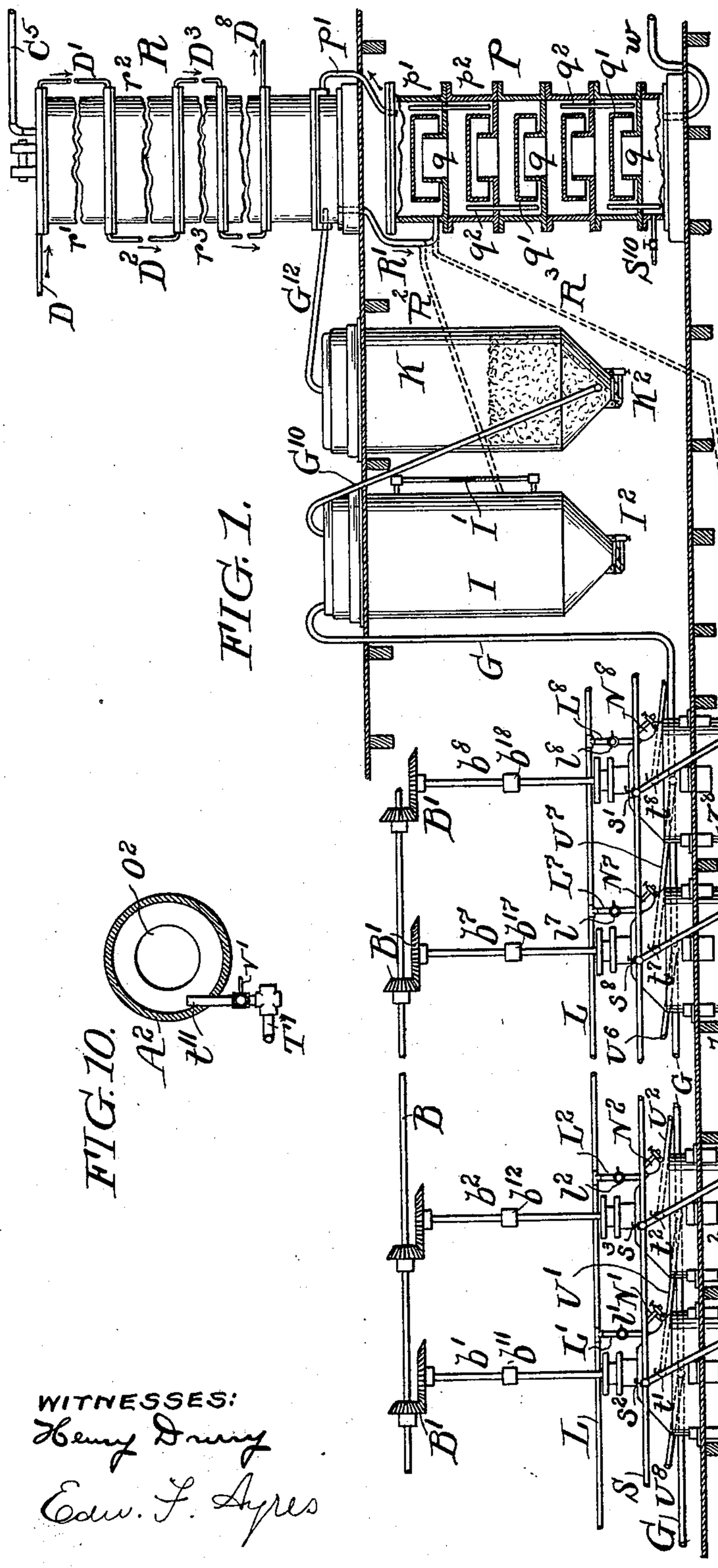

FIG. 1.

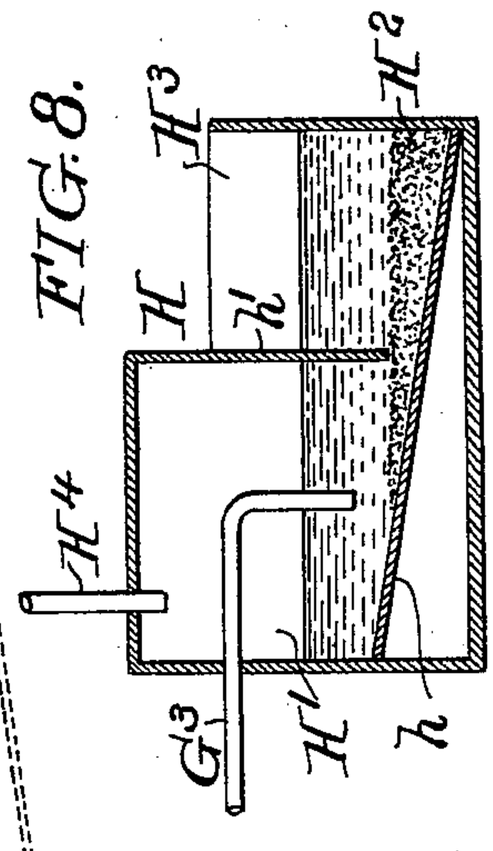

FIG. 8.

FIG. 10.

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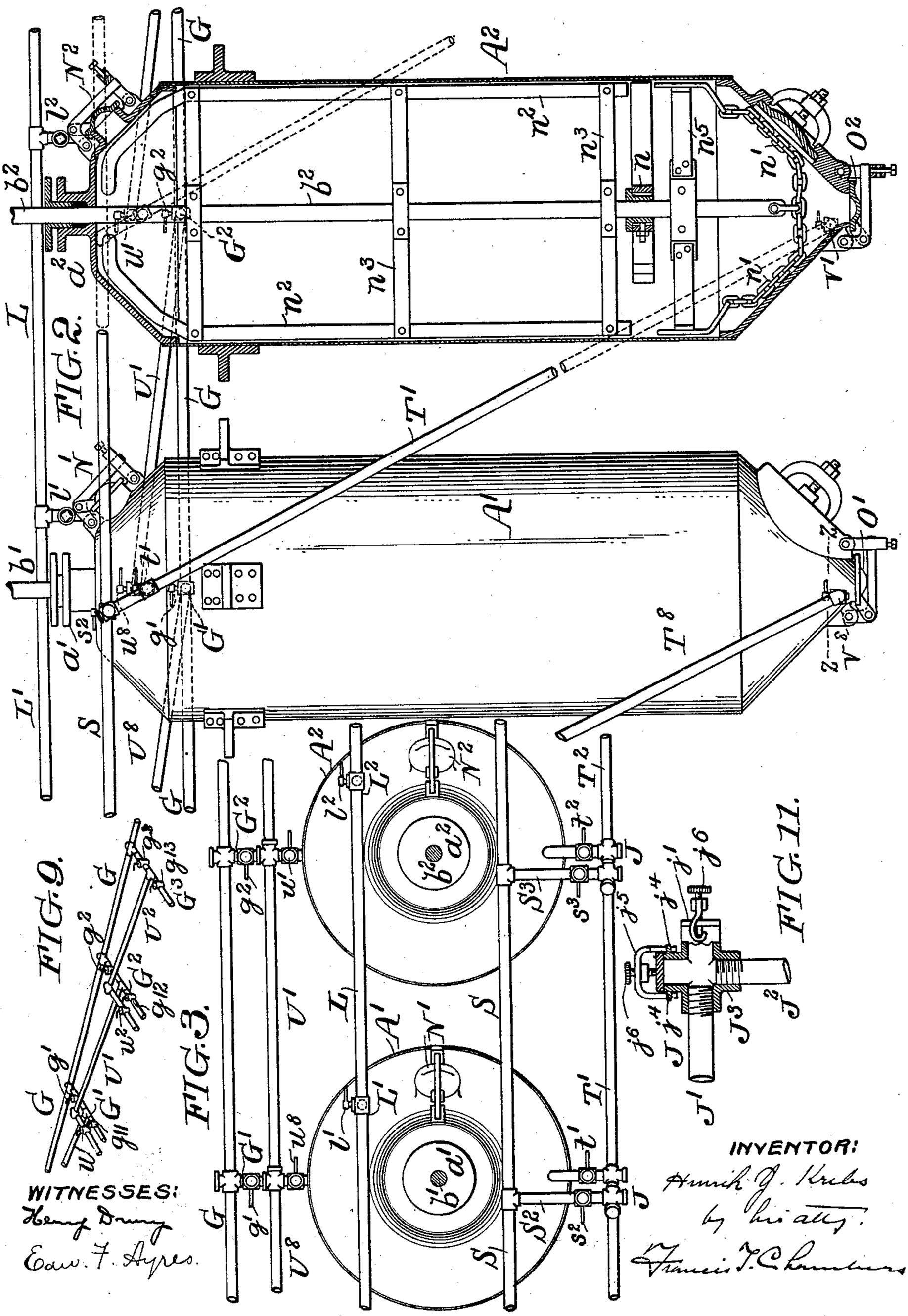

FIG. 2.

FIG. 11.

FIG. 9.

FIG. 3.

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FIG. 4.

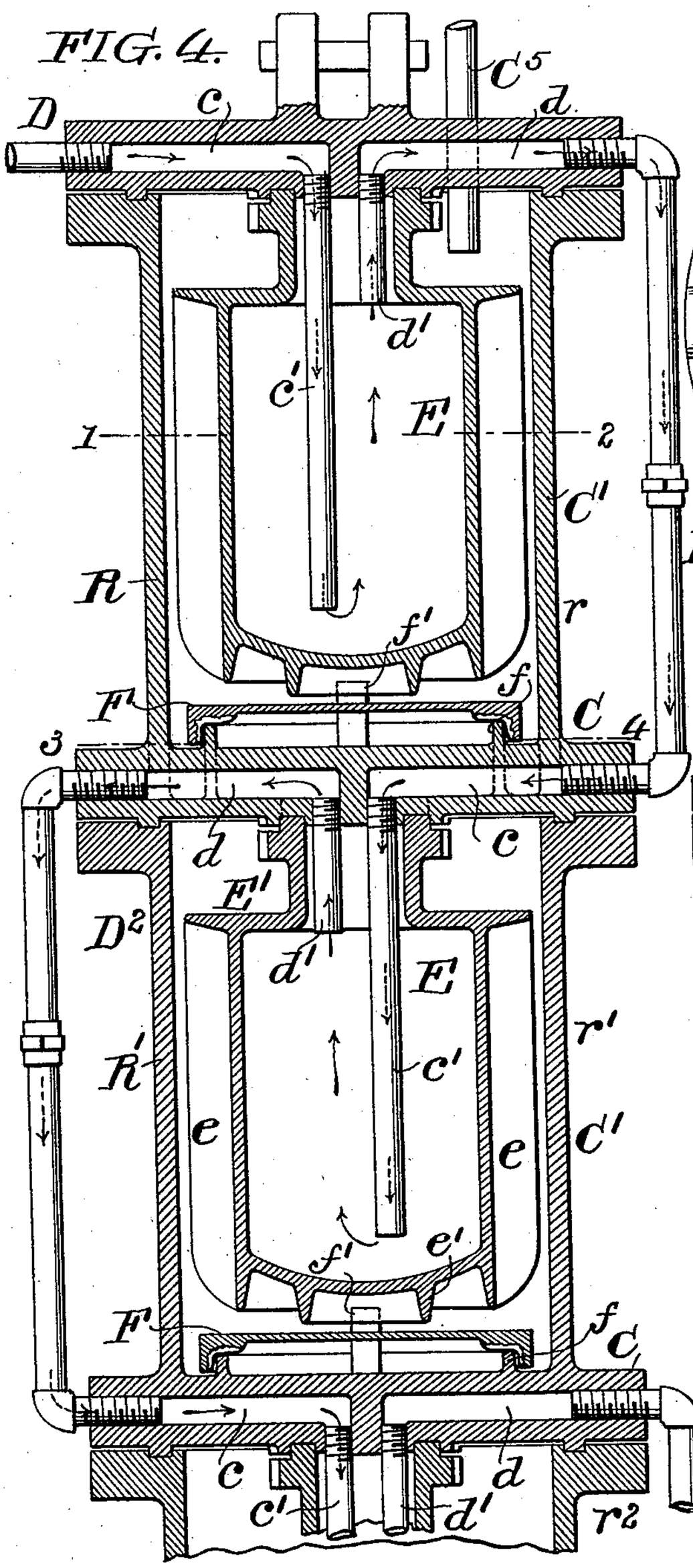

FIG. 5.

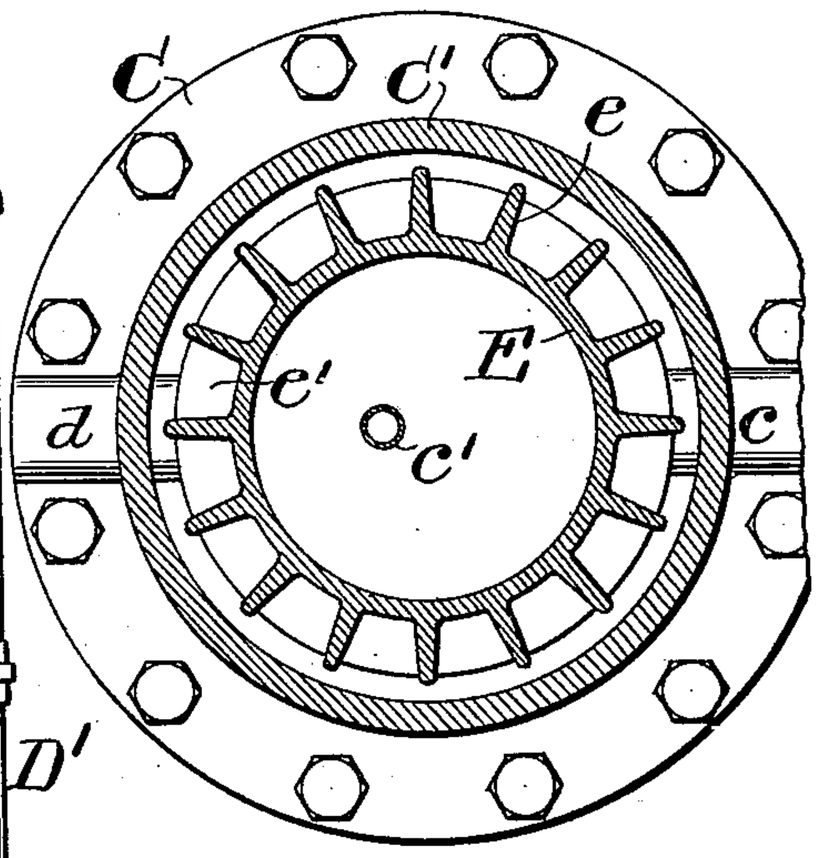

FIG. 6.

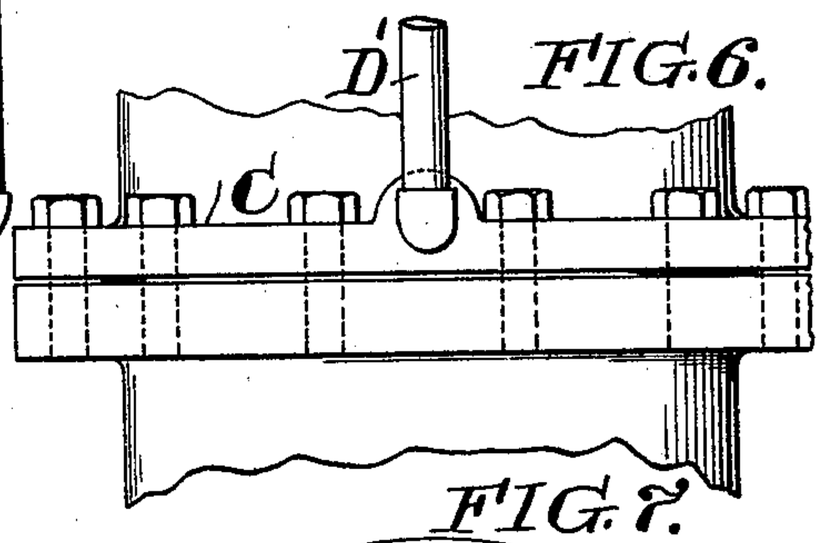

FIG. 7.

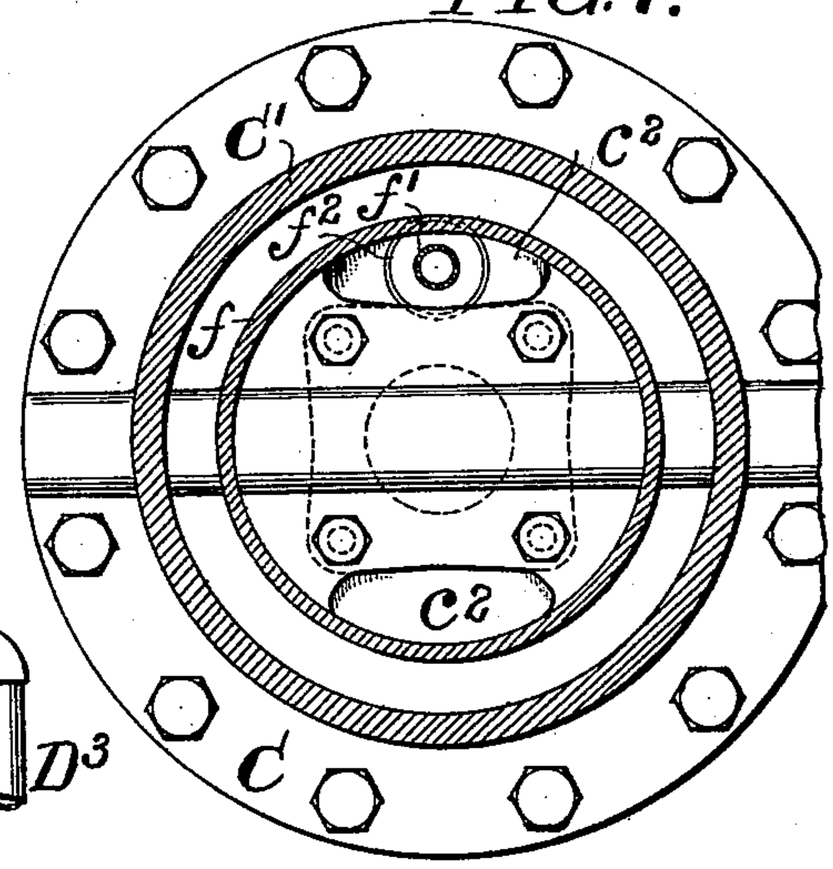

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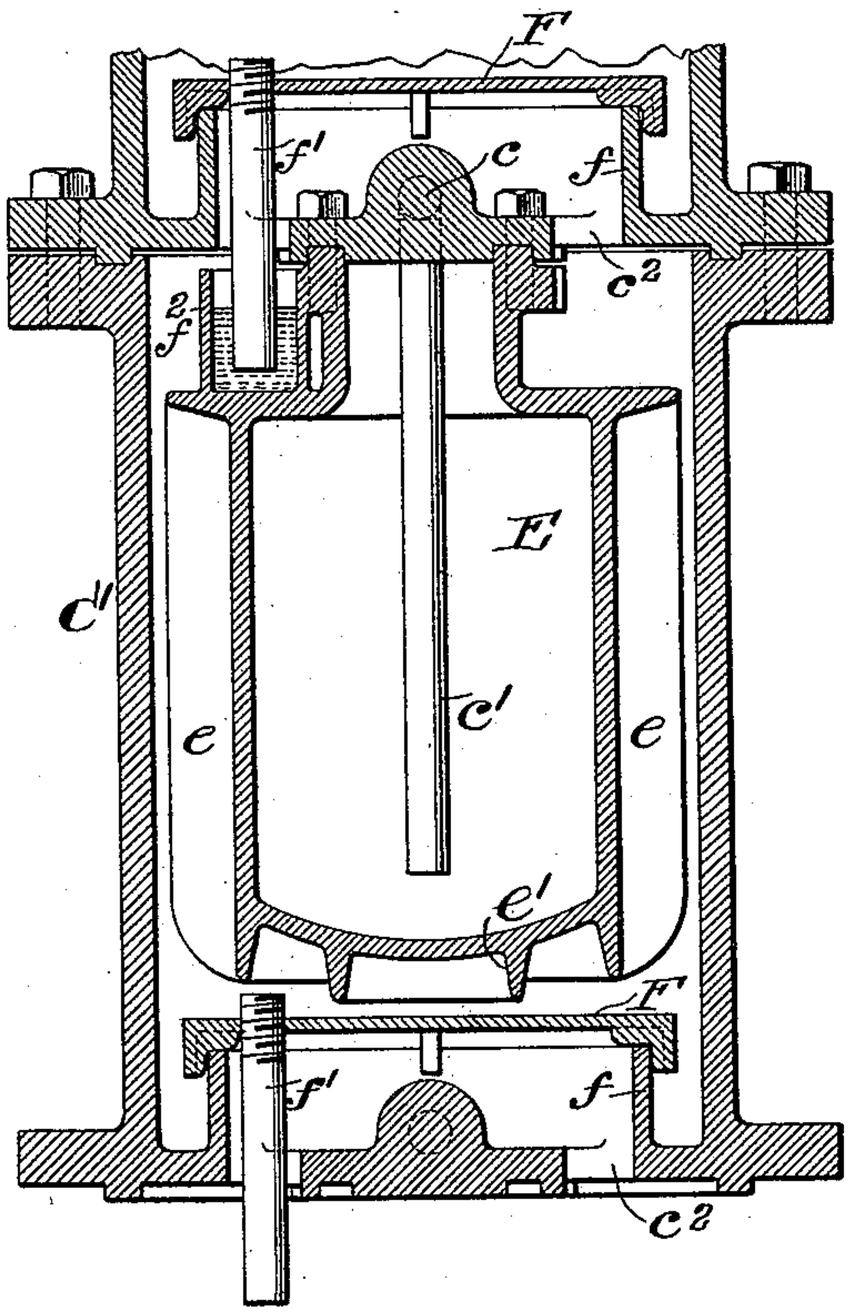
Fig. 12.



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UNITED STATES PATENT OFFICE.

HENRIK J. KREBS, OF WILMINGTON, DELAWARE.

DISTILLING APPARATUS.

SPECIFICATION forming part of Letters Patent No. 589,000, dated August 31, 1897.

Application filed June 1, 1894. Serial No. 513,114. (No model.)

To all whom it may concern:

Be it known that I, HENRIK J. KREBS, a citizen of the United States, residing at Wilmington, in the county of New Castle, in the State of Delaware, have invented certain new and useful Improvements in Distilling Apparatus, of which the following is a true and exact description, reference being had to the accompanying drawings, which form a part thereof.

My invention relates to a distilling apparatus, and more particularly to an apparatus designed to separate ammonia from the substances carrying it.

The main object of my invention is to provide an apparatus whereby ammonia-gas can be separated from the substances carrying it continuously and whereby the gas obtained will be of nearly uniform richness.

Heretofore it has been common to introduce steam into the bottom of a still containing some ammonia-carrying substance and carry away the steam laden with whatever gas it takes up as it rises through the contents of the still from the top of this still either to a suitable rectifier or, in some cases, to a second and third still and then to a rectifier. In such cases, however, steam rapidly impoverishes the charge in the first still, or in all the stills if several are used, and the resulting gas is very poor, it being necessary, however, in order to separate all the ammonia, to continue sending steam through the apparatus. Finally, when the gas is all separated the steam must be turned off and the vessels dumped and recharged. These difficulties are overcome by the use of my improved apparatus, wherein the distilling operation can be carried out continuously.

My invention mainly consists in arranging a series of stills for ammonia distillation in an endless circuit by suitable pipe connections passing from the top of each still to the lower part of the next and providing a take-off pipe connected to each still and cocks in the several pipe connections, whereby any or all of the connections can be closed at will.

My invention further consists in various details of construction and combinations of parts which are more specifically pointed out in the claims, which are appended to and form part of this specification.

My invention will be best understood as explained in connection with the accompanying drawings, in which—

Figure 1 is a side view of a distilling apparatus arranged according to my invention. Fig. 2 is an enlarged view of two of the stills shown in Fig. 1 and their connections, one of said stills being in sections. Fig. 3 is a plan view of two of the stills, showing their connecting-pipes. Fig. 4 is a vertical section of a part of the rectifying-column. Fig. 5 is a section on the line 1 2 of Fig. 4. Fig. 6 is a fragmentary elevation of the column shown in Fig. 4. Fig. 7 is a section on the line 3 4 of Fig. 4. Fig. 8 is a sectional view of a vessel for the production of ammonia salts. Fig. 9 is a detail view of some of the pipe connections not clearly shown in Fig. 1. Fig. 10 is a cross-section on the line 2 2 of Fig. 2. Fig. 11 is a detail view of a pipe-joint which is very useful in my improved apparatus; and Fig. 12 is a vertical section of a part of the rectifying-column, taken at right angles to the section of Fig. 4.

A' A^2, &c., are stills, which are arranged in an endless series by means of pipe connections T' T^2, &c., which lead from the upper part of each still to the lower part of the next still in series, it being noticed that the pipe operating with a particular still is marked by a symbol having the same index as that still.

The pipes T' T^2, &c., are provided with cocks or valves v' v^2, &c., so that any of the pipe connections can be opened or closed at will.

G is a gas or take-off pipe, which is provided with connections G' G^2, &c., leading to the stills A' A^2, &c., cocks or valves g' g^2 being adapted to close these connections.

S is a pipe for introducing steam into the stills, though any other suitable source of heat may be employed, if desired. This pipe S has branches S' S^2, with valves s' s^2 leading to the several stills. This connection I conveniently make through the connecting-pipes T' T^2, &c., since when steam is being turned into a particular still the preceding still in the series would ordinarily be open to the take-off or gas pipe, and to prevent steam from entering this still from which gas is being taken off I provide valves t' t^2, &c., in the connections T' T^2, &c., between the stills and the place where the pipes T' T^2, &c., are entered by the pipes S' S^2, &c. The opera-

tion of such a series of stills is easily understood. The top of one still is connected to the lower part of the next, and so on for any number of stills, the last one used being connected to an off-take pipe. Heat is applied, as by turning steam into the stills, and the operation is continued till the charge in the first still has become weak. This still is disconnected, a new charge introduced, the outtake pipe connected to this newly-charged still, and connection made so that it will be the last still in the series, when the action is repeated. To adapt this series for use in manufacturing ammonia from ammoniacal liquor, I provide a liquor-pipe L, having branches L' L^2, &c., leading to the various stills l' l^2, &c., being valves in said pipes whereby this ammoniacal liquor can be conducted into any of the stills.

N' N^2 are openings in the tops of the stills, through which lime and water or undissolved ammonia salts can be introduced, and O' O^2 are dumping-holes, through which the charge can be removed when the gas is exhausted therefrom.

B is a shaft having bevel-gear B', which serves to operate shafts b' b^2, &c., which are connected to stirrers in the stills, preferably through suitable clutches b^{11} b^{12}, &c., to prevent breakage. These stirrers preferably consist, as shown in Fig. 2, of a frame or frames n^2, extending close to the sides of the still, and a chain n', secured to a portion of the frame and adapted, when the stirrer is turned, to drag on the bottom of the still.

n is a bearing for the shaft, as b^2, which carries the stirrer-frame, and may be arranged at the bottom of the shaft or on a spider, as shown.

In order to insure that there shall be a thorough stirring of the charge and that all portions thereof shall be subjected to the heating action of the steam, I preferably arrange the pipes T' T^2, &c., which conduct steam or steam and gas into the stills, so that their nozzles, as t^{11}, (see Fig. 10,) will project into the stills more or less tangentially, as shown, so that the effect will be to cause a rotation of the liquor and other ingredients in the still. I preferably turn the stirrers in the same direction as the rotation caused by the flow of the steam and gas, so that the whole mass is kept in constant agitation.

The gas-pipe G in the arrangement shown in Fig. 1 leads into a vessel I, which serves to intercept spray and other impurities which the gas may carry with it.

I' is a glass to indicate the height of liquid in the vessel I, and I^2 is a discharge-outlet closed by a suitable lid. From the vessel I the gas may be conducted, as by a pipe G^{10}, to a vessel K, charged with a suitable caustic alkali, such as caustic soda, to intercept impurities, as carbonic acid, contained in the gas, and from the chamber K a pipe G^{12} serves to conduct the gas to a rectifying-column.

If it is not desired to form aqua-ammonia, but some salt, the gas can be conducted directly from the stills to a suitable vessel, as II, (see Fig. 8,) which contains the acid with which the gas unites to form a salt. In the particular arrangement shown the vessel II is provided with a sloping bottom h and has a partition h', dividing it into two chambers II' and II^2. The gas enters at G^3 and unites with the acid to form an ammonium salt, which crystallizes out and collects at the lower slope of the bottom, from which it is removed through the open top II^3.

II^4 is a pipe to permit the escape of any excess of gas.

The vessels I K, while useful, may be dispensed with, particularly when the gas is to be employed to make a salt, and I have provided connections, such as are best seen in Figs. 2, 3, and 9, whereby any of the stills can be employed as a vessel to catch the spray. U' U^2, &c., are these connections and connect each still with the next one in series and are shown very conveniently opening at one end into the gas-pipe sections G' G^2, &c., so that one tapping of the still may be dispensed with. g^{11} g^{12} and u' u^2 are valves which serve to control the flow of gas through these pipes. As best shown in Fig. 9, the sections U', &c., tap the stills at a higher point than the gas-pipes, and if it is desired to use one still to catch the spray the gas, with the spray carried thereby, would pass through a section, as U^8, the valves u' g^{12} being open and g^2 being closed, and would discharge into the still A^2 through the section G^2 and the spray would be deposited in said still, while the gas would escape through the section U^3 G^3 into the gas-pipe, the valves u^2 and g^3 being open and g^{13} closed.

Referring now to the rectifying-column, R is a rectifier, which is preferably made up of a number of sections r r'. These sections consist of a casing, as C', which has within it a closed bell or chamber E, which forms part of a cold-water conduit. The conduits leading to and from this bell E are conveniently formed by passages c d, cast in the cover-plate C of the casing, the inlet-passage c being supplemented by a pipe c', which leads the entering fluid near to the bottom of the bell, the water escaping from the bell through pipe d' and passage d. The pipe d' only extends a short way down into the bell, preferably below the neck E' thereof, so that the air remaining in the neck will form an air-cushion and prevent water-hammers, and so that in case water should be left in the rectifier and should freeze there will be room for its expansion without breaking the bell.

D is a water-inlet leading preferably to the top section, and D' D^2 are pipes connecting the different sections, so that the cooling water after passing through the bell in one section is conducted to the bell in the next.

D^8 is the final escape-pipe for the water.

It will be noted that the cover C for one section serves as the base-plate for the one

next above it, and the plate is provided with an opening or openings c^2, whereby the gas and steam in one section can ascend to the next. This opening or openings c^2 is preferably surrounded by a flange f, which is covered by a bell F, so that the gas rising from one section into the next will be forced to bubble up through a layer of liquid, and to permit the passage of liquid I preferably provide a pipe f', which is secured to the bell F and which projects into a cup, as f^2, in the section below, so as to seal the pipe. It is, of course, evident that the bell F may be dispensed with, if desired, and then the water will fall through the same passages c^2 through which the gas rises.

To provide the bell E with a larger cooling-surface and incidentally to center it, I conveniently provide it with wings, as e e', and C^5 is the final escape-pipe, through which the ammonia-gas escapes, freed from steam.

The liquid resulting from the condensation is carried away by the pipe R', and may be conducted, if desired, into the vessel I or into one of the stills by means of suitable pipes, (indicated by dotted lines at R^2 R^3,) but I prefer to lead it into an analyzer-column P. The analyzer is also made into sections p' p^2, &c., each of which, except the top section, connects with the section above through a flanged opening q, which is covered by a bell q'. A pipe q^2 also extends through and to some distance above the floor of each section, so as to permit the flow of water after a desired amount has accumulated.

S^{10} is a steam-pipe which leads into the lowest section, and w is a trapped pipe for conducting off the water. The liquid resulting from the condensation in the rectifier therefore flows into the analyzer P, where it meets the steam introduced through pipe S^{10}, which drives off any gas which may be carried down by said water, this gas rising into the rectifier through the pipe P'.

In order to permit the easy cleaning of the pipes, particularly the still connections, which are apt to be clogged with lime or other impurities, I prefer to form the points J of the pipes in the manner shown in Fig. 11, where J^3 is a cross-shaped coupling.

J' J^2 represent pipes connected to two adjacent coupling-arms, while the arms opposite to the pipes are closed by plates j', which may be easily removed when it is necessary to clean the pipes.

Ordinary screw-caps cannot be advantageously used, since the lime and carbonates of which the charges largely consist make a very hard scale and fill up the threads. I therefore form lugs, as j^4, on the pipes, with which hooks on a bow j^5 engage. A screw j^6 passes through the bow and bears on a flat plate, as j'. Packing may be placed between the plates and the end of the pipes to make a tight joint.

In operating the distilling apparatus to obtain ammonia-gas a suitable amount of ammonical liquor is permitted to flow into each still through the liquor-pipe L, and also lime, if necessary, is put in through the openings N', &c. The proper valves are opened and the steam is turned on. It enters the bottom of one still, liberates ammonia-gas, and passes with the gas to the bottom of the next still, and so on, finally emerging from the last still of the series laden with a rich gas. The impurities are separated in the vessels I and K and the steam condensed in the rectifier-column. After the steam has been turned on sometime in the first still the gas is practically all driven off from the charge therein, and this still is dumped and a new charge placed in it and the steam turned on into the next still in the series, as explained, the gas being taken off from each still and the steam turned into each still of the series successively, thus permitting a continuous action and insuring that the gas will always be of substantially the same strength when it finally comes off from the series of stills.

It may here be noted that while I much prefer to operate the stills in the manner just described—that is, in series—the arrangement of stills substantially as shown in Fig. 1, but omitting the connections which permit me to employ them as an endless series, is a great improvement over the usual construction, permitting me to charge each still independently and employ a few or many stills, as may be desirable. In regard to the construction of the rectifier it will also be noted that the chamber or bell E is suspended from the plate or cover C, which forms the partition between the adjoining sections of the rectifier, a tight joint being made between the neck of the bell and the partition-plate. It will be further noted that the inlet and outlet conduits for the cooling medium have no joints in the main portion of the rectifier, all the joints being in the neck of the bell. I also prefer to provide each of the vessels with a pressure-gage, as Z' Z^2, &c., which will indicate if and where a stoppage in the pipe occurs.

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

1. In a distilling apparatus the combination with one or more independent stills of a series of condensing-chambers R and a series of distilling-chambers P arranged as described so that the condensed liquid from the gas condensed in the chambers R will flow down into the chambers P and the gas and vapors from chambers P will rise into chambers R and a delivery-pipe leading from the independent stills to the condensing-chambers R.

2. In a distilling apparatus for ammonia the combination with a series of stills connected in an endless circuit by pipes leading from the head of each still to the lower part of the still next in consecutive order, cocks for closing any of the pipes at will, a take-off

pipe having connections with each still and cocks arranged in each take-off pipe connection, a series of condensing-chambers forming a rectifier R and into which the take-off pipe leads, and a series of distilling-chambers forming an analyzer P arranged relatively to and connected to the rectifier R substantially as and for the purpose specified.

3. The combination with a series of stills connected in an endless series by pipes as T' T^2 leading from one still into the lower part of the still next in order, cocks in said pipes, a take-off pipe G having branches leading from each still, and pipe-sections U' U^2 &c. leading from each still to the next and suitable cocks in said pipe-sections all substantially as specified, and so that any of the stills may be used as a chamber to catch spray carried by the gas.

4. A condenser consisting of a number of sections as *r r'* &c. each section consisting of a casing C', a cover C having passages *c d*, a bell or chamber E in said casing into which the passages *c d* open, the passages *c d* and bell E forming part of a conduit for a cooling medium all arranged substantially as specified and so that the cover of one section forms the bottom of the next.

5. A condenser consisting of a number of sections *r r'* &c. each section consisting of a casing C', a cover C having passages *c d*, a bell or chamber E into which the passages *c d* open, a pipe as *c'* connected to the opening and extending to near the bottom of the bell E, all arranged substantially as and for the purpose specified.

6. A rectifying-column having sections *r r'* each consisting of a casing C', a bell E in said casing, a cover C having passages *c d* and pipes *c' d'* extending different distances into the bell.

7. A rectifier-column having sections *r r'* &c. each section consisting of a casing C', a bell E having a neck E', a cover C having passages *c d*, a pipe *c'* extending to near the bottom of the bell and a pipe *d'* extending substantially to the base of the neck substantially for the purpose specified.

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