

(No Model.)

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APPARATUS FOR DISTILLING AMMONIA.

No. 259,146.

Fig. 1.

Patented June 6, 1882

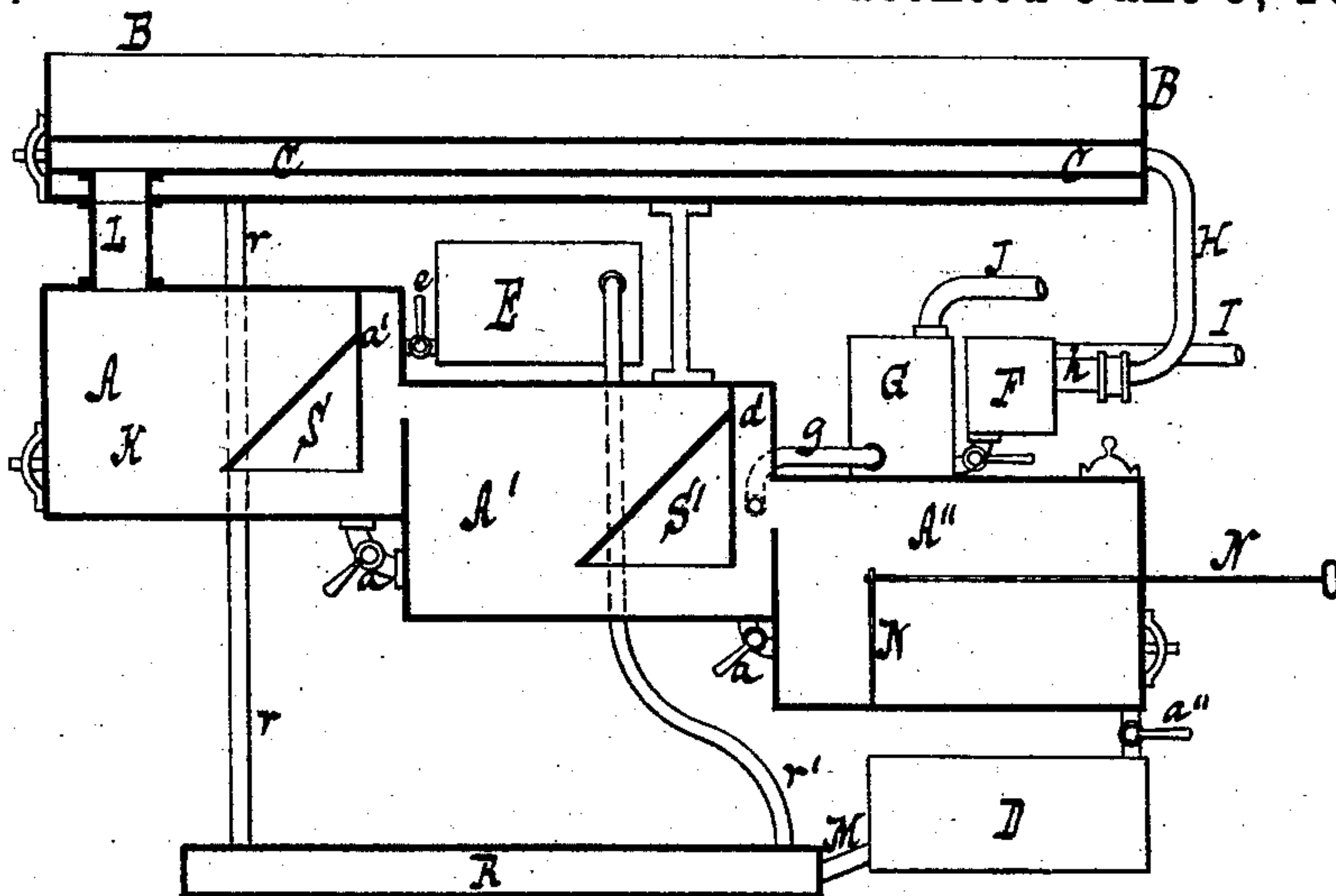


Fig. 2.

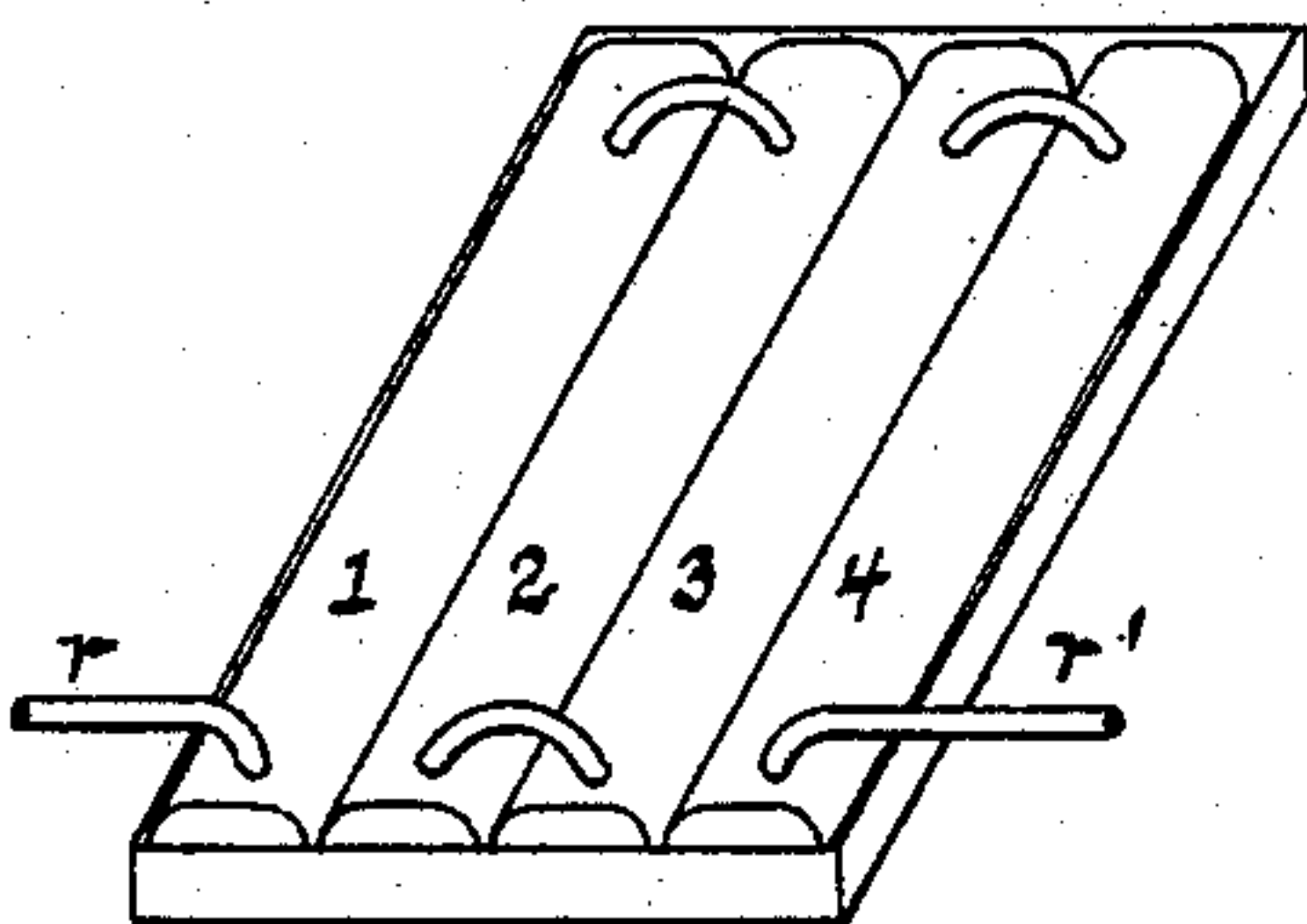
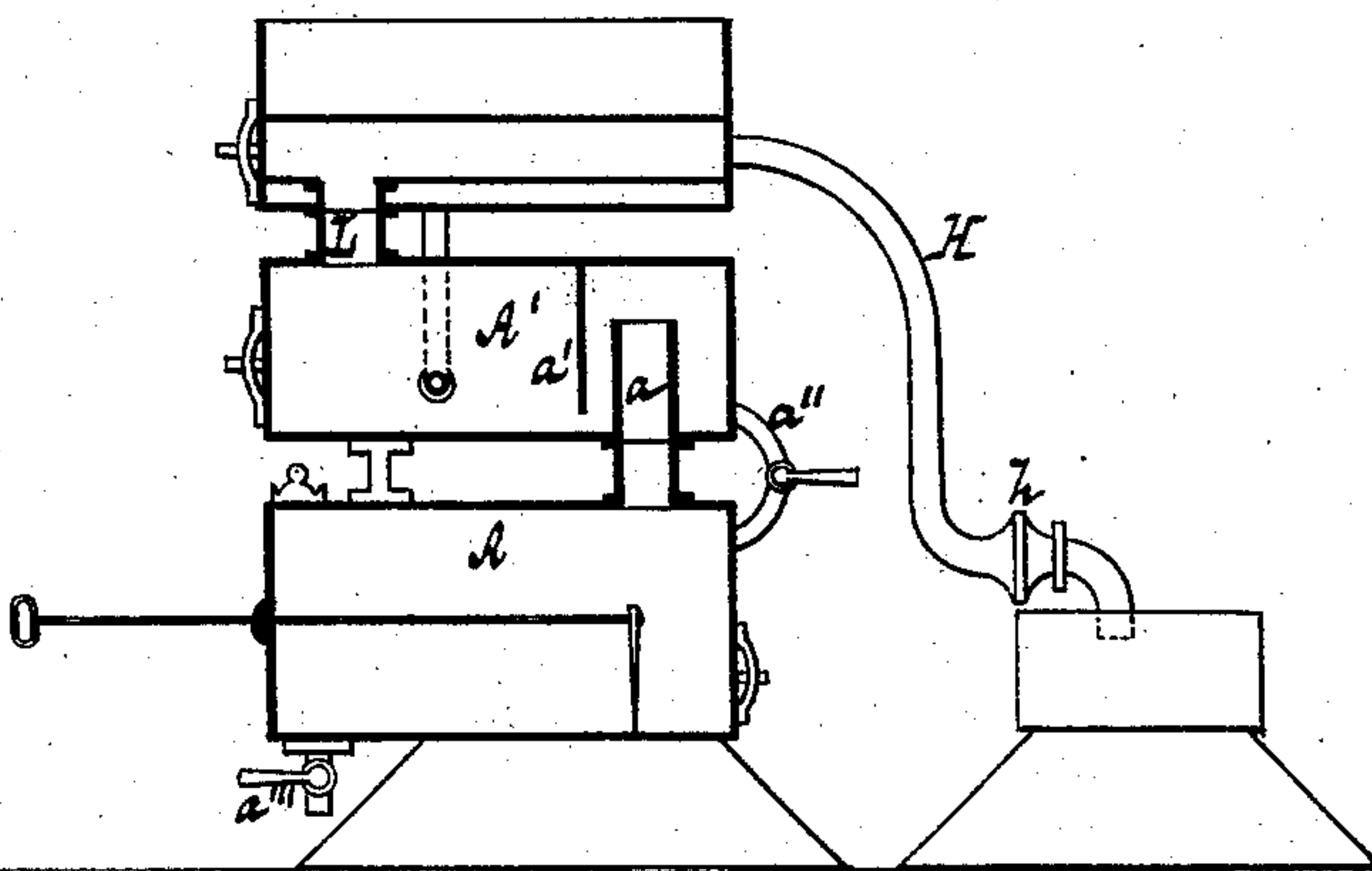


Fig. 3.



Inventors.

Witnesses

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# UNITED STATES PATENT OFFICE.

HENRI JOSEPH ERNEST HENNEBUTTE, OF ANGLET, AND CHARLES JUST FÉLIX RAOUL DE JANNEL MENARD, VICOMTE DE VAURÉAL, OF BIARRITZ, FRANCE, ASSIGNORS TO THE SOCIÉTÉ ANONYME DES PRODUITS CHIMIQUES DU SUD-OUEST, OF PARIS, FRANCE.

## APPARATUS FOR DISTILLING AMMONIA.

SPECIFICATION forming part of Letters Patent No. 259,146, dated June 6, 1882.

Application filed December 1, 1881. (No model.) Patented in France January 15, 1879, No. 128,575, and in England July 19, 1880, No. 2,964.

*To all whom it may concern:*

Be it known that we, HENRI JOSEPH ERNEST HENNEBUTTE and CHARLES JUST FÉLIX RAOUL DE JANNEL MENARD, Vicomte de Vauréal, citizens of the Republic of France, residing, the former at Anglet, the latter at Biarritz, in the Department of the Basses Pyrénées and Republic of France, have invented new and useful Improvements in Apparatus for Distilling Ammoniacal and other Liquors, of which the following is a specification.

This invention relates to an improved apparatus for distilling ammoniacal liquors, consisting of a receiver into which such liquors are led, retort communicating with said receiver; and a condenser communicating with said retort, said condenser being provided with a tube for carrying off the vapors.

The retort is preferably made in several compartments communicating with each other, some of the compartments being provided with partitions and inverted-bell-shaped receivers for catching the gaseous vapors and forcing them through the liquid, thus keeping the liquid in agitation.

The ammoniacal vapors, on coming from the condenser, may be led through driers or drying-cases for removing the moisture which has been carried along in the distillation.

The chief advantages secured by this apparatus are, first, treatment of the ammoniacal liquors in thin layers and in partial vacuum, with constant agitation, secured without the aid of mechanical force; second, dephlegmation or concentration between 80° to 90° centigrade of the greater part of the ammonia and of the ammoniacal salts, which are set free at this temperature, while the emission of aqueous vapor is almost nothing; third, reheating the liquors by the emitted vapors as well as by the residuary waters, whereby saving of fuel is secured; fourth, dispensing with all apparatus liable to become choked—such as worms—the arrangement being simple and durable in its nature and adapted to be cleaned without any cessation of the work.

This invention is illustrated in the accompanying drawings, in which Figure 1 represents a side view. Fig. 2 shows a detached view of the heating-case. Fig. 3 shows the apparatus modified so as to occupy less space.

Similar letters indicate corresponding parts.

The retort may be of varying dimensions and of any suitable shape, preferably rectangular. As shown in Fig. 1, it is divided into three compartments, A A' A'', the compartments being situated on different levels, and partitions rising about ten centimeters from the bottoms of the higher compartments prevent the liquids therein from flowing into the next lower one. From the roofs of the compartments A A' are shown depending partitions *a'*, carrying inverted-bell-shaped receivers S S'. The partitions *a'* reach below the surfaces of the liquid in the compartments A A' and compel the gaseous vapors passing through the retort to force their way through the liquid and agitate the same. The receivers S S' catch the gaseous vapors, which vapors, when they fill the receivers S S', force their way out and through the liquid, thus securing additional agitation. By means of cast faucets *a* the emptying of the liquids from one compartment into the other may be secured.

Above the retort is placed a rectangular open case, B, serving as a receiver, into which the waste waters or sluice-waters are led. In this receiver lies the condenser C, so that the warmth of this condenser C is absorbed by the waters in the receiver B, which thus serves as a cooler. The condenser C catches the vapors from the retort, which flow into the condenser through the tube L. The waters in the receiver B are heated by contact with the condenser C to about 30° centigrade, and then flow through the tube *r* and through the boilers 1 2 3 4, Fig. 2, in the heating case or chest R, where they are further heated to about 80° or 90° centigrade by the residuary or devaporized waters which have been operated on, and which flow from the cleanser D through the pipe M into the case R, which case is preferably



made closed, and may be provided with a vent or faucet for emptying the same. From the boiler 4 the waters rise through the tube *r''* into the reservoir E, from whence they are allowed to flow into the retort by opening the faucet *e*. In the retort the ammoniacal vapors are expelled from the waters by the application of heat, which vapors flow into the condenser C, as already stated. A thermometer, K, is secured to the retort. The waters, when devaporized or freed from ammonia, are allowed to flow into the cleanser D.

In the compartment A, where the waters lose about four-fifths of their ammonia, the distillation presents the following characteristics: The ammoniacal vapors set free in the compartment A' accumulate under the receiver S, and take the place of the liquids in A, and impart to them a wave-like motion, which has for its object to agitate the same. The said ammoniacal vapors are transformed by contact with the liquid into ammonia very feebly hydrated, which flows off through the connecting-tube L, while the vapor of water is condensed in the liquid, the temperature of which is never made to exceed 90°.

The extremely rich ammoniacal vapors coming directly from the vapors in compartment A, or in the compartment A' after dephlegmation in A, pass into the tubular condenser C, the walls of which are maintained at a relatively low temperature by the water in the receiver B. The ammoniacal vapors diminishing in volume, a corresponding quantity of outer air would enter into the apparatus; but a sluice-valve, *h*, situated at the base of the tube H, allows the escape of vapors which might be in excess in the apparatus, while it prevents absolutely the entrance of air, and thus a relative vacuum is established in the tubular condenser C. It thus follows that the ammoniacal liquids contained in the compartment A are distilled between 80° and 90° centigrade in relative vacuum, while a constant agitation is obtained without mechanical force. It thus follows that the ammoniacal vapors from the compartment A' are dephlegmated in A. In said compartment A' the temperature of the waters attains 100° and the separation of the ammonia is almost perfect. In A'', under the influence of milk of lime, the waters lose their last traces of ammonia.

A ball-valve at the top of compartment A'' serves at the same time as safety-valve and as funnel for the introduction of the lime.

A rake, N, passing through a stuffing-box, furnishes the means for cleaning the compartment A'' without stopping the operation.

Above the compartment A'' are two small cases or chests, F and G. In the case or chest F are stored up the waters carried along during the distillation, while the dry ammonia-gas passes through the escape-tube I into the acid bath. From F the concentrated ammoniacal waters are allowed to pass into the small case or chest G, the bottom of which is formed

by the upper wall of the compartment A''. Under the influence of the high temperature of this compartment the concentrated liquids lose their ammonia, which is received by the acid bath. This ammonia is freed by the escape-tube J. Both the tubes I and J lead into an acid bath, in which the ammonia is neutralized and forms a salt.

The waste concentrated liquids are led by the escape-pipe *g* into the compartment A'. Acting on the residuary waters, they flow through the faucet *a''*. Then they lose their lime by standing at rest in the cleansing apparatus D, and finally, passing through the pipe M, they pass successively each compartment of the heater R, where they lose gradually their heat, which is received by the fresh sluice-waters which flow through the boilers, as before explained.

We construct for certain special industries small simple apparatus which are rather a reduction in size than a modification of the above-described apparatus. (See Fig. 3.) A', retort where is obtained the dephlegmation between 80° and 90°; A, retort where the distillation is completed, which is provided with a ball-valve and a rake, and also an escape-pipe, *a'''*, for emptying the compartment. The ammoniacal vapors in A are freed and pass through the large tube *a*. Then they pass under the partition *a'*, entering below the surface of the liquid contained in A', imparting to it a wave motion. Then the ammoniacal vapors pass up through the communicating tube L into the condenser C, where, in consequence of the refrigeration, they diminish in volume. The sluice-valve *h*, situated at the base of the escape-pipe H, hindering the entrance of the air into the apparatus, a relative vacuum is established. In these small apparatus the distillation is thus effected under the same conditions as in the large ones. The compartments A A' in the small apparatus also communicate with each other by a tube, *a''*, closed by a valve. In this small apparatus, on account of the narrow space, the receivers S S' may be dispensed with, the partition *a'* insuring sufficient agitation of the liquids.

Of course the apparatus need not be confined to the distillation of ammoniacal liquors, but may be adapted to any use for which it is serviceable.

The condenser C, as also one or several of the compartments of the retort, may be provided with firmly-closing doors or removable plates, so that access to the interior may be obtained.

What we claim as new, and desire to secure by Letters Patent, is—

1. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, of a retort communicating with said receiver, said retort being divided into several compartments, and a condenser, C, having a tube or other means for carrying off the vapors, substantially as set forth.



2. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, and condenser C, of a retort, said retort being divided into compartments communicating with each other and provided with partitions  $a'$ , substantially as and for the purpose set forth.

3. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, and a condenser, C, of a retort, said retort being divided into compartments communicating with each other and provided with partitions  $a'$ , having inverted-bell-shaped receivers S S', substantially as set forth.

4. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, of a heating-chest, R, a retort, and a condenser, C, substantially as set forth.

5. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, of a heating-chest, R, reservoir E, a retort communicating with said reservoir, and a condenser, C, substantially as described.

6. In an apparatus for distilling ammoniacal and other liquors, the combination, with a

receiver, B, of a heating-chest, R, a retort, a cleansing apparatus, D, communicating with said retort, and a condenser, C, substantially as set forth.

7. In an apparatus for distilling ammoniacal and other liquors, the combination, with a receiver, B, and a retort communicating with said receiver, of a condenser, C, and drying-chests F G, communicating with said condenser, substantially as set forth.

8. A retort consisting of two or more compartments, A A' A'', communicating with each other, and provided with partitions  $a'$ , having inverted-bell-shaped receivers, substantially as described.

In testimony whereof we have hereunto set our hands and seals in the presence of two subscribing witnesses.

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