

(No Model.)

3 Sheets—Sheet 1.

H. GRÜNEBERG & E. BLUM.  
APPARATUS FOR DISTILLING AMMONIA.

No. 358,196.

Patented Feb. 22, 1887.

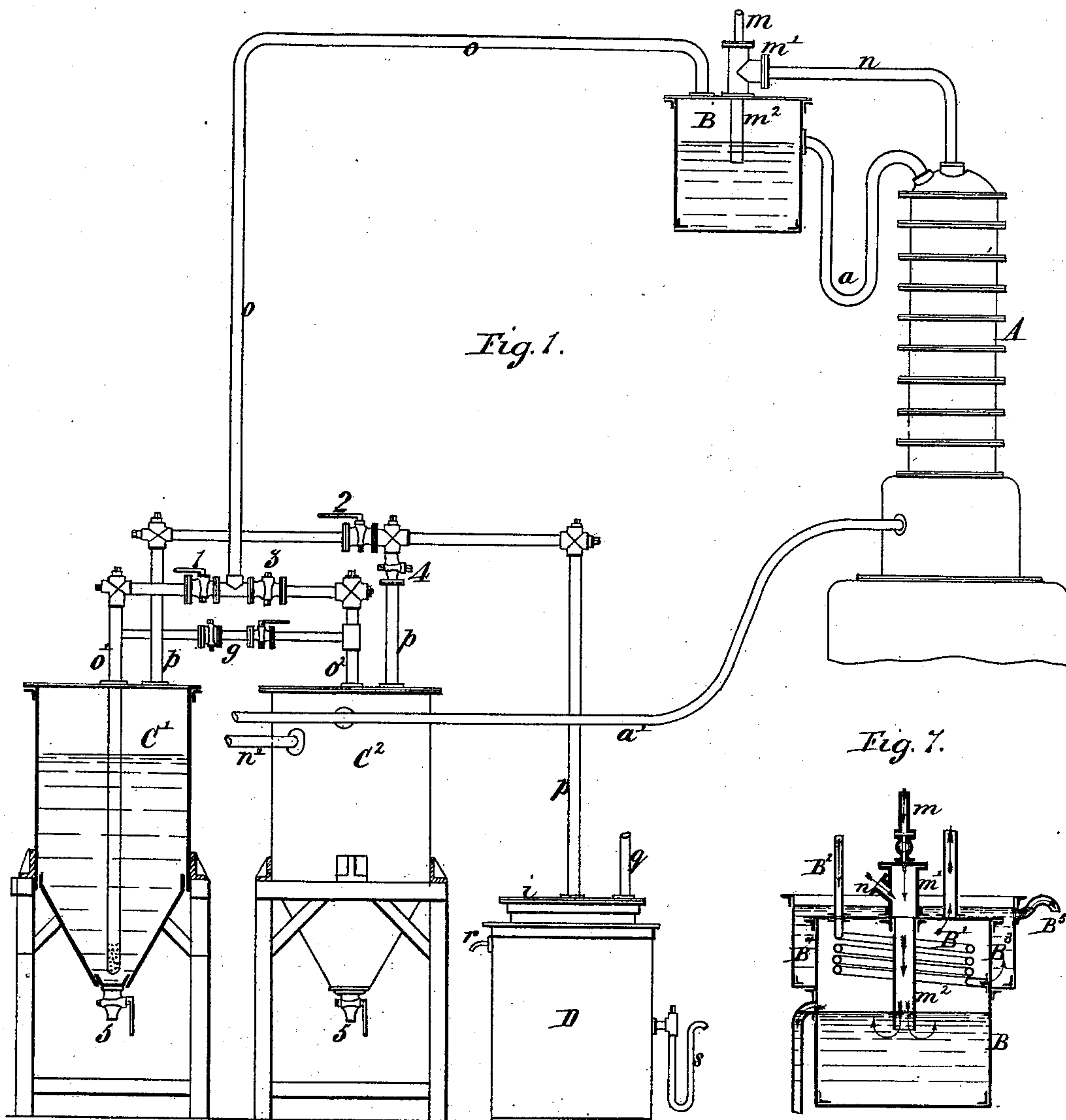


Fig. 1.

Fig. 7.

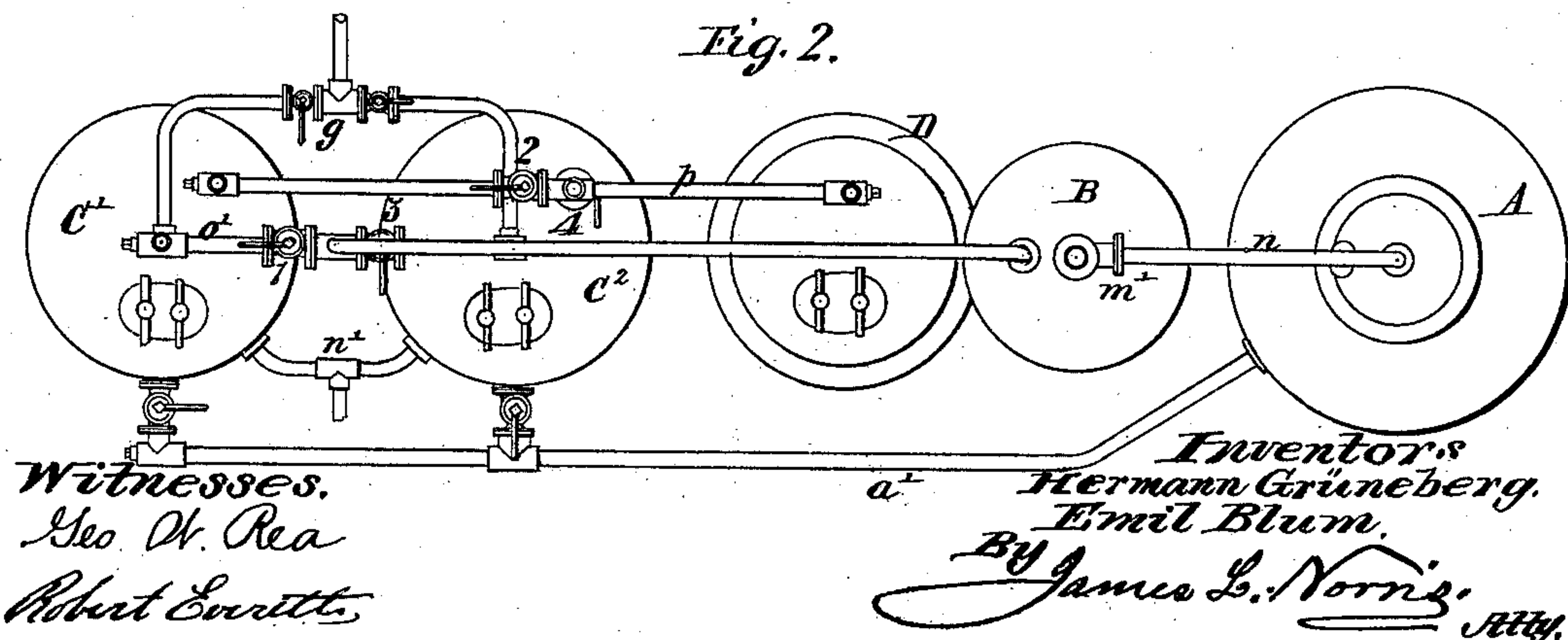


Fig. 2.

Witnesses.  
Geo. W. Rea  
Robert Corliss

Inventors  
Hermann Grüneberg.  
Emil Blum.  
By James L. Norris, atty.

(No Model.)

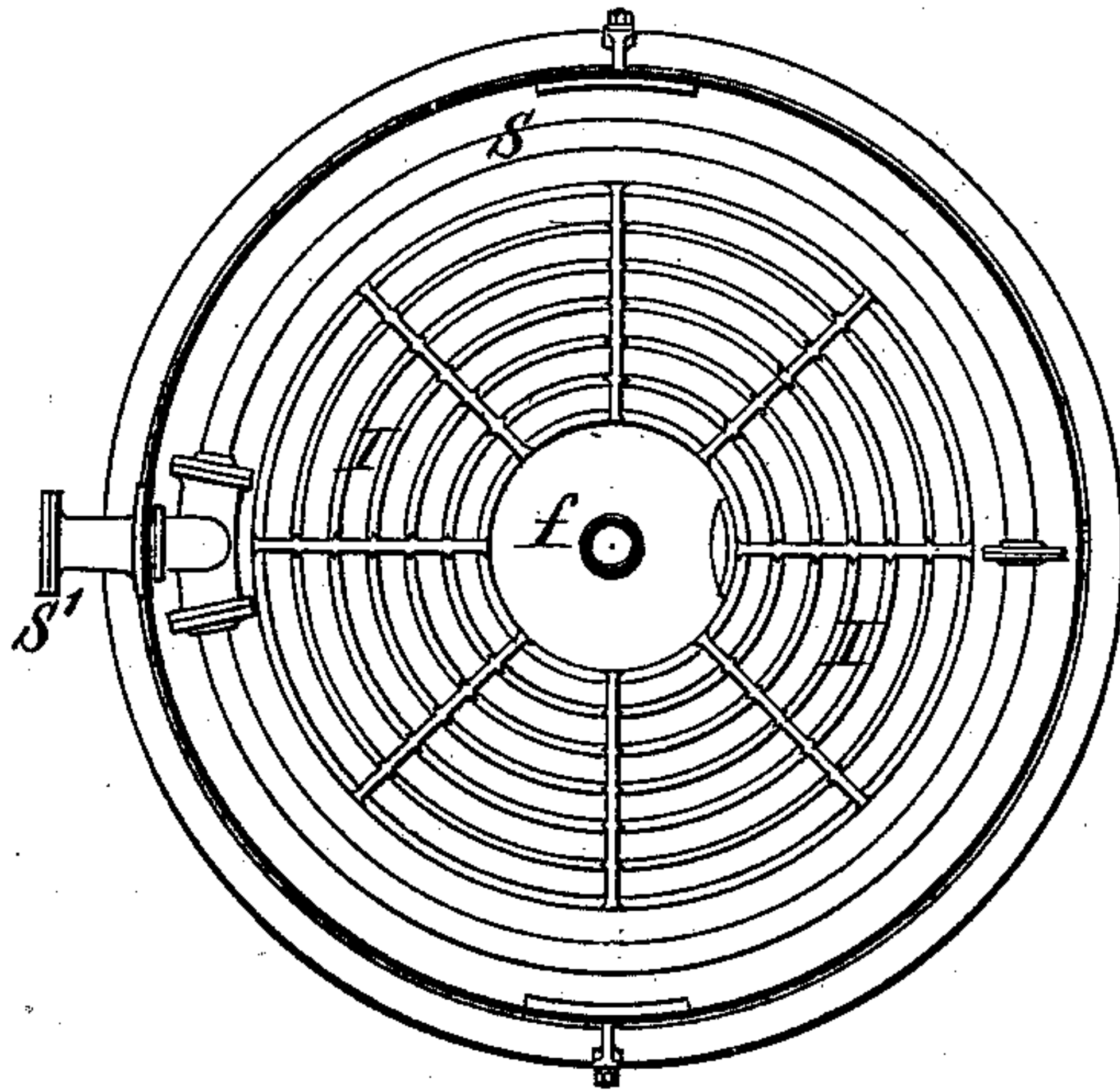
3 Sheets—Sheet 2.

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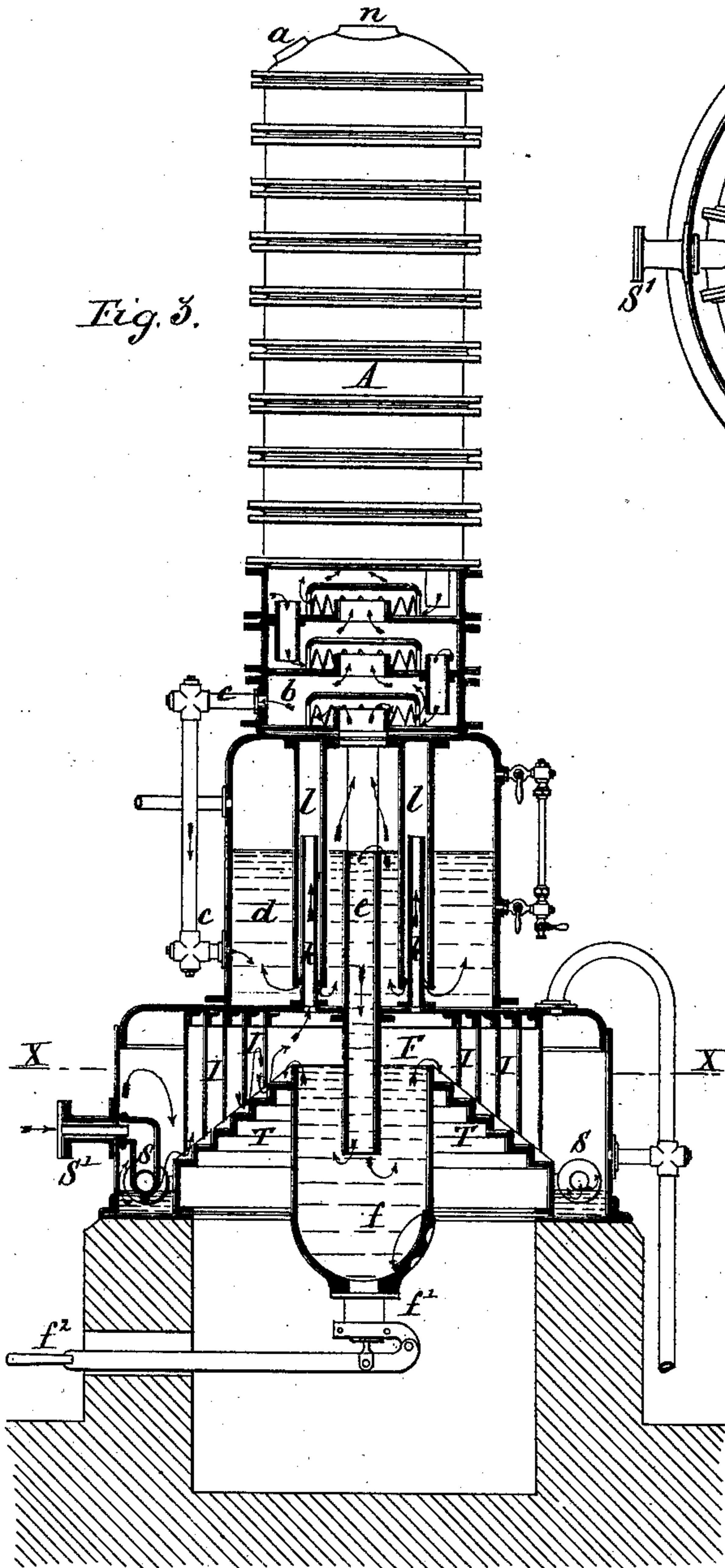
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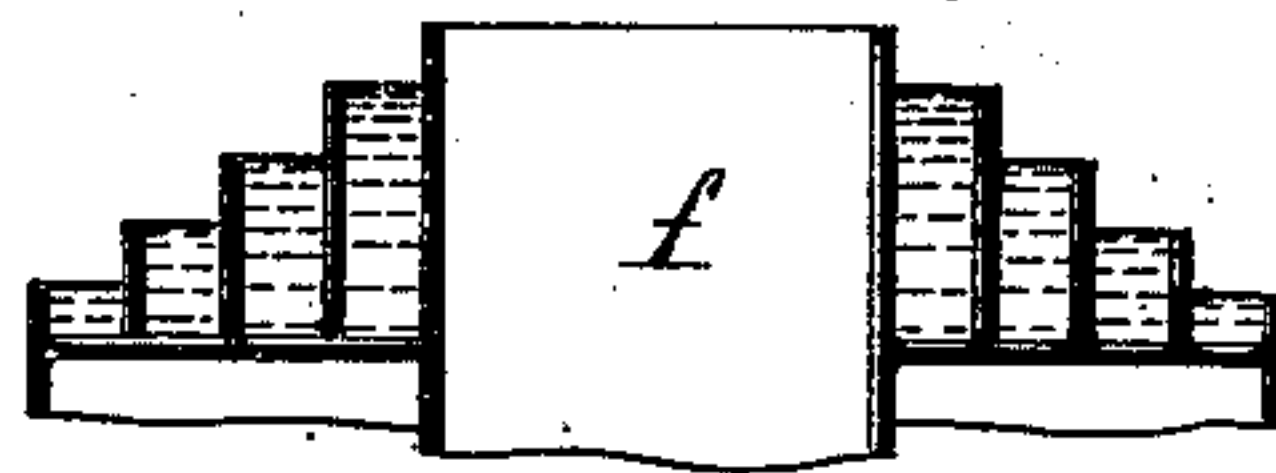
*Fig. 4.*



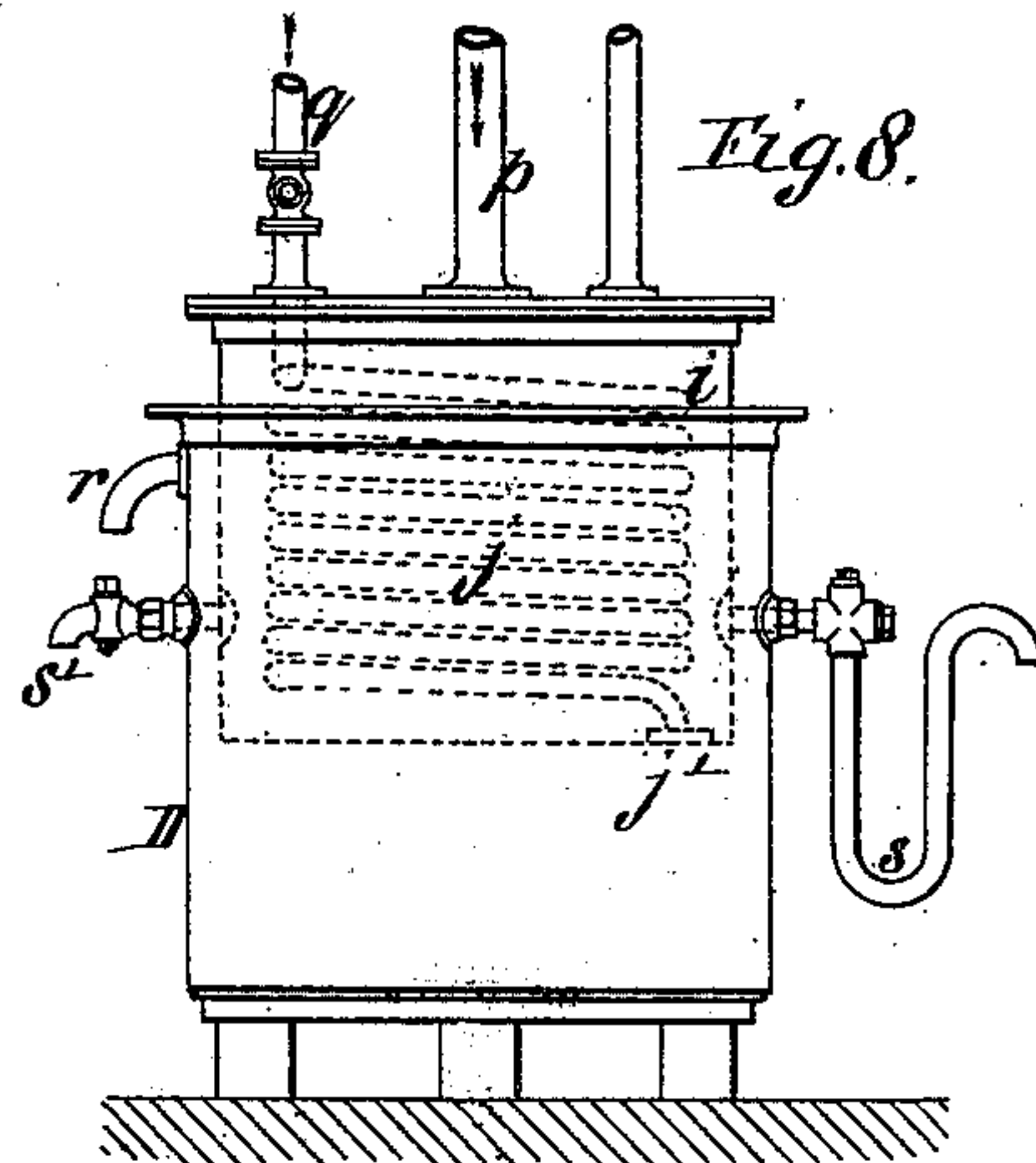
*Fig. 3.*



*Fig. 5.*



*Fig. 8.*



*Witnesses.*  
*Geo. H. Rea*  
*Robert Corbett.*

*Inventors*  
*Hermann Grüneberg*  
*Emil Blum*  
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*Atty.*



(No Model.)

3 Sheets—Sheet 3.

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APPARATUS FOR DISTILLING AMMONIA.

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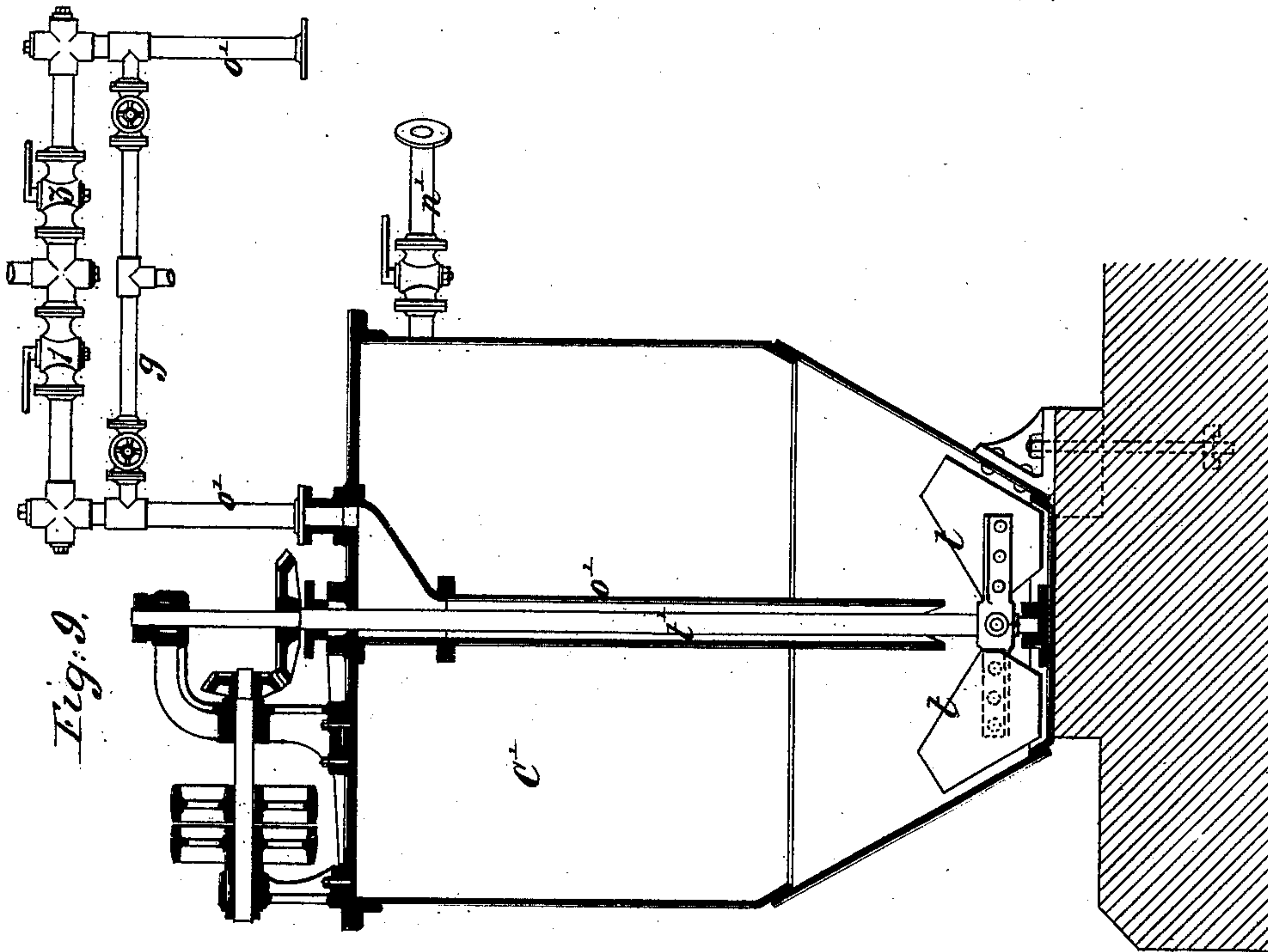


Fig. 9.

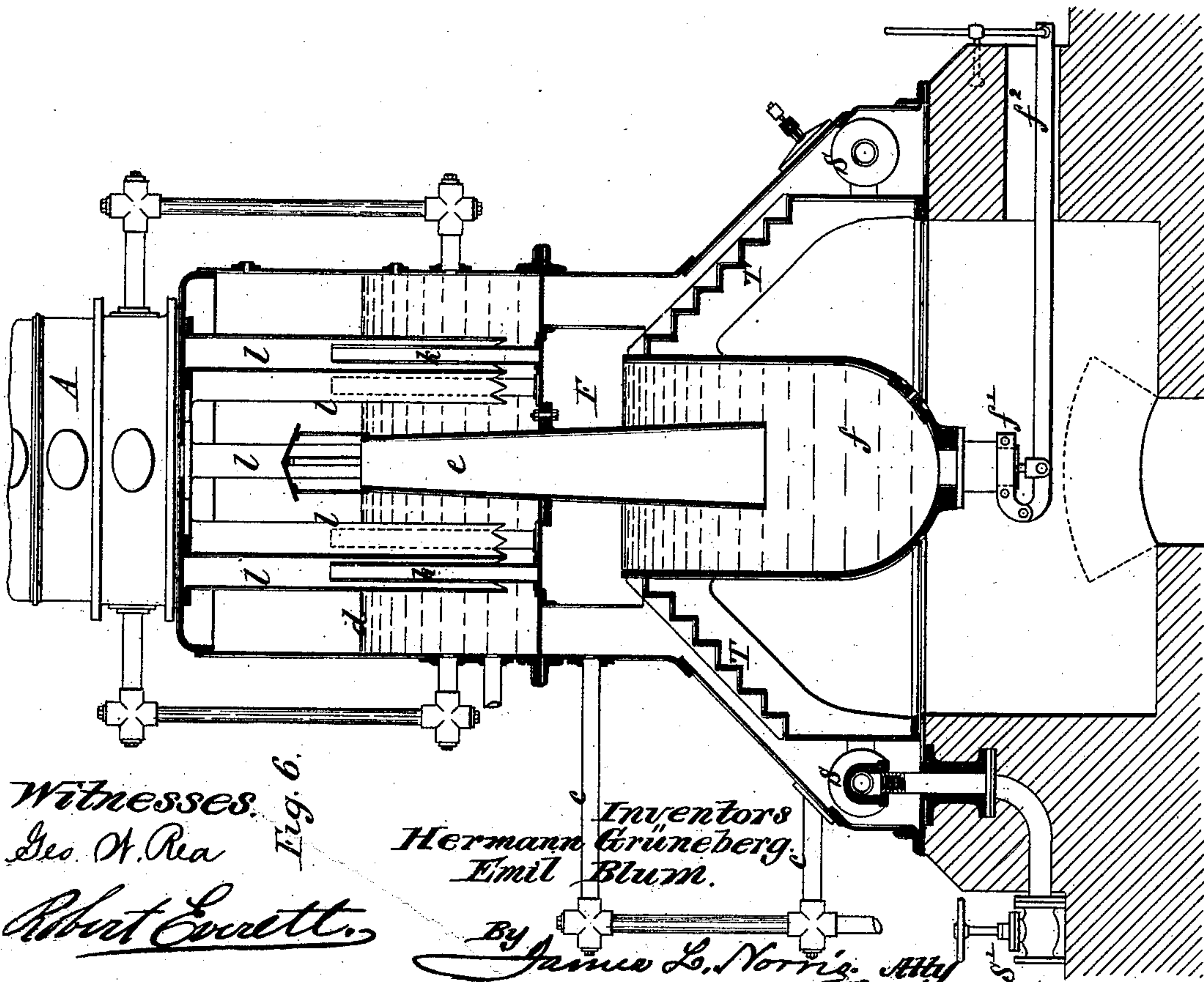


Fig. 6.

Witnesses.

Geo. H. Rea

Robert Everett.

Inventors  
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By James L. Norris, Atty



# UNITED STATES PATENT OFFICE.

HERMANN GRÜNEBERG, OF COLOGNE, AND EMIL BLUM, OF BERLIN, GERMANY, ASSIGNORS OF ONE-THIRD TO HENRY SIMON, OF MANCHESTER, ENGLAND.

## APPARATUS FOR DISTILLING AMMONIA.

SPECIFICATION forming part of Letters Patent No. 358,196, dated February 22, 1887.

Application filed April 9, 1886. Serial No. 198,385. (No model.) Patented in Germany February 11, 1885, No. 33,320; in England February 25, 1885, No. 2,578; in France April 11, 1885, No. 168,211; in Austria-Hungary April 13, 1885, No. 14,324, and in Italy April 14, 1885, XIX, 18,201.

*To all whom it may concern:*

Be it known that we, HERMANN GRÜNEBERG and EMIL BLUM, subjects of the Emperor of Germany, residing, respectively, at Cologne, in the German Empire, and Berlin, in the German Empire, have invented new and useful Improved Apparatus for the Distillation of Ammonia from Ammoniacal Liquors and for the Treatment of the Resulting Ammoniacal Vapors, (for which we have obtained Letters Patent in Germany, February 11, 1885, No. 33,320; Great Britain, February 25, 1885, No. 2,578; France, April 11, 1885, No. 168,211; Austria-Hungary, April 13, 1885, No. 14,324, and Italy April 14, 1885, Vol. XIX, No. 18,201,) of which the following is a specification.

This invention relates to apparatus for distilling and concentrating ammoniacal liquors in a continuous manner, wherein such liquors are caused to pass downward through a column wherein, as they flow successively from one tray to the next below, they are brought into intimate contact with steam passing in a contrary direction, whereby the steam is made to liberate the ammonia from the liquor, the latter being then made to flow into a chamber containing milk of lime, whereby any ammonia remaining in a combined state is rendered free. The ammoniacal vapors and steam that issue from the top of the still are first made to pass into a vessel, where they are brought in contact with the ammoniacal liquor before it passes into the still, so that on the one hand this liquor becomes heated preparatory to its distillation, while on the other hand, the ammonia-vapors become cooled. These are then conducted into one or other of two vessels charged with milk of lime, in order to free them from carbonic acid and other impurities, the said vessels being so arranged that while the ammonia-vapor is passing through the one the contents of the other, which have previously had the ammonia-vapor passed through them, are first treated with steam, in order to drive off the ammonia taken up by the lime-water, and are then discharged, after which the vessel is charged with a fresh sup-

ply of milk of lime. The ammonia-vapor issuing from these vessels is lastly passed into a concentrating-vessel for the production of concentrated ammoniacal liquor, or into apparatus for the production of either sal ammoniac or sulphate of ammonia.

We will proceed to describe the above-mentioned improved apparatus in connection with the accompanying drawings, in which—

Figure 1 shows a diagram elevation, partly in section, of the entire apparatus. Fig. 2 shows a plan, and Fig. 3 shows a sectional elevation, of the still. Fig. 4 shows a cross-section of the same at *xx*, Fig. 3. Figs. 5 and 6 show modified constructions of the bottom part of the still. Fig. 7 shows a modified construction of the combined heater and cooler. Fig. 8 shows the concentrating-vessel for the ammonia-vapor, and Fig. 9 shows a modified construction of the lime-vessels.

The still consists of the well-known arrangement of a column, *A*, in which the ammoniacal liquor is caused to flow downward successively through a number of trays, while steam is caused to flow upward in intimate contact with liquor, so as to liberate the ammoniacal vapor therefrom. Arrived at the bottom tray, *b*, more or less deprived of its ammonia, the liquor flows through the pipe *c* into the chamber *d*, containing milk of lime, so that by mixing with this any ammonia remaining in the liquor in a combined state is rendered free. The liquor, after treatment with the milk of lime, which is continuously fed into the chamber, overflows through the pipe *e* and descends into a well, *f*, in a second chamber, *F*. In this well any solid suspended matter becomes deposited, while the liquor overflows its edge onto the stepped surface *T*, consisting of a series of circular steps of increasing diameter, so that the liquor in flowing down over them becomes spread out in layers of gradually-decreasing depth until at the lowest step it is in an exceedingly fine state of subdivision, and flows at a very slow speed. Below the lowest step is a circular perforated pipe, *S*, into which steam is supplied through the branch *S'*, and the steam issuing thence is caused by the con-



centric annular partitions I to pass upward in intimate contact with the liquid flowing down over the steps, so as to take up what free ammonia is still contained therein. Arrived at the top of the steps, the mixed steam and ammonia-vapor passes into the upper chamber, *d*, through tubes *k*, situated concentrically within other tubes, *l*, closed at their upper ends and open at the lower ends, so that the mixed steam and vapor, after rising through the tubes *k*, has to descend again through *l* and issue in a finely-divided state into the mixture of milk of lime and liquor contained in the chamber. The steam is thus caused to disengage a further quantity of ammonia from the liquor, and then passes up through the tower A in the well-known manner, finally issuing at top, together with the ammonia-vapor, through the pipe *n*. The deposit in the well *f* is discharged from time to time through the discharge-valve *f'*, operated by the lever *f''*.

Instead of constructing the stepped surface T as shown at Fig. 3, it may consist of a number of concentric rings of graduated height, as shown in section at Fig. 5. It may also be made of polygonal or segmental form instead of circular; also, in place of the annular partitions I, the casing of the apparatus may be formed inclined, as shown in the modification at Fig. 6, so as to cause the steam to pass in close contact with the liquid.

The pipe *n* conducts the mixed steam and ammoniacal vapor into a vessel, B, into which the ammoniacal liquor that is to be fed into the still is also caused to enter through a pipe, *m*, opening into the same box, *m'*, into which the mixed steam and ammoniacal vapors pass, so that the liquor and vapor are brought into intimate contact and issue together through the pipe *m''* into B, where the vapor is made to bubble up through the liquor on its way to the pipe *o*. The ammonia-vapor thus becomes effectually cooled, while at the same time the liquor becomes heated before flowing off through the bent pipe *a* into the still. Fig. 7 shows a modified arrangement of the above-described combined heater and cooler, wherein the upper part of the vessel B contains a coil, B', through which cold water is made to flow, entering at B<sup>2</sup> and issuing at B<sup>3</sup>, into a tank, B<sup>4</sup>, surrounding B, whence it overflows through a pipe, B<sup>5</sup>. By this means the ammonia-vapor contained in the upper part of the vessel B is still further cooled before issuing through *o*. The cooled ammoniacal vapor is conducted from the vessel B through the pipe *o* to the one or other of the two vessels C' C<sup>2</sup> containing milk of lime, through which the vapor is made to pass so as to free it from any carbonic acid, &c. For this purpose the pipe *o* is provided with two branches, *o'* *o''*, leading, respectively, into the vessels C' C<sup>2</sup>, in which they descend to near the bottom, where they are perforated, so as to allow the ammonia-vapor to issue in divided streams into the lime-liquid. Each branch pipe is provided

with a stop-cock, 1 and 3, so that while the ammonia-vapor is being admitted into the one vessel the communication with the other one can be cut off. From the tops of the vessels C' C<sup>2</sup> other pipes, *p*, also provided with stop-cocks 2 and 4, pass either to a cooling apparatus, D, for the production of concentrated ammoniacal liquor, or with the necessary apparatus of known construction for the production of either sal ammoniac or sulphate of ammonia.

The construction of the vessel D is shown more clearly in the enlarged elevation at Fig. 8. It consists of a casing, D, within which is a vessel, *i*, into which passes the pipe *p* from the lime-vessels, and which contains a coil, *j*, for the circulation of cold water, entering at top through the pipe *q* and issuing at bottom *j'* into the casing D, whence it passes away at *r*. The ammoniacal vapor, entering the vessel *i* from the pipe *p* and becoming refrigerated and condensed by contact with the coil *j*, flows off through the bent pipe *s*.

*s'* is a cock for the discharge, from time to time, of accumulated oily impurities floating on the ammonia-liquor.

When the milk of lime in the one vessel—say C'—has become spent by the passage of ammoniacal vapor through it, the cocks 1 and 2 are closed and 3 and 4 are opened, so that the ammoniacal vapor will now pass from *o* into C<sup>2</sup> and thence to the cooler D. A steam-pipe, *g*, provided with branches and stop-cocks, communicates with the pipes *o'* *o''* of the vessels C' C<sup>2</sup>, so that after C' has been cut off from the supply-pipe *o* steam is admitted into the milk of lime, so as to drive off any ammonia taken up thereby, the resulting mixture of ammoniacal vapor and steam being led back into the still A through a pipe *a'*, also provided with stop-cocks. The spent lime-liquor is then discharged from C' through the discharge-cock 5, and a fresh charge of milk of lime having been introduced through the pipe *n'*, C' is again put in communication with *o* and D, and the communications of C<sup>2</sup> are closed so soon as the contents hereof have become spent. Thus the continuous operation of the still and the purifying and condensing apparatus is maintained.

Fig. 9 shows a vertical section of a somewhat modified form of the vessels C' C<sup>2</sup>, in which these are provided with stirring apparatus *t*, consisting of revolving inclined vanes situated immediately below the orifice of the pipe *o'*, through which the ammoniacal vapor enters, so that an intimate admixture of the latter in a finely-divided state with the milk of lime is insured. The shaft *t'* of the vanes passes up through the pipe *o'* and is rotated through gearing by any convenient motor.

Having thus described the nature of our invention and the best means we know for carrying the same into practical effect, we claim—

1. In combination with apparatus for dis-



tilling ammonia from ammoniacal liquor, a chamber containing a stepped surface onto which the lime-liquor charged with ammoniacal vapor flows from the lime-chamber above, the liquor on flowing down in a thin stream over the stepped surface being subjected to intimate contact with steam, whereby the ammonia is disengaged therefrom and carried back into the lime-chamber, substantially as herein described.

2. In combination with a stepped surface over which the liquor containing ammonia descending from the ammonia-still is made to flow, a well or pocket into which the liquor flows from the lime-chamber before passing on to the stepped surface, in order to separate solid suspended matter therefrom, substantially as herein described.

3. The combination, with a stepped surface over which the liquor containing ammonia descending from the ammonia-still is made to flow, of annular partitions or equivalent appliances for causing the stream to come in contact with the liquor flowing over the steps, substantially as herein described.

4. Apparatus for treating ammonia-vapor passing from the ammonia-still with milk of lime, consisting of two closed vessels having each, first, a pipe branching from the pipe conveying the ammonia-vapor from the still and provided with a stop-cock; secondly, a steam-supply pipe communicating with the said branch pipes and also provided with a stop-cock; thirdly, another branch pipe provided with a stop-cock communicating with a

pipe for conveying the ammonia-vapor to the condensing-vessel or other apparatus; fourthly, a branch pipe provided with a stop-cock communicating with a pipe for conveying the ammonia-vapor liberated by the action of the steam back into the still; fifthly, a pipe for charging the vessel with milk of lime, and, sixthly, a cock for discharging the spent milk of lime, all arranged and operating substantially as and for the purposes herein set forth.

5. Apparatus for concentrating ammonia, consisting of a closed vessel surrounded by a jacket for the circulation of cold water and containing a coil of pipes through which the cold water is made to flow before entering the jacket, the ammonia-vapor being led into the top of the vessel by a pipe, so as to become condensed by contact with the coil and the cooled sides of the vessel, the resulting concentrated ammonia-water being led away through a siphon-bend, substantially as herein described.

In testimony whereof we have signed our names to this specification, in the presence of two subscribing witnesses, the 8th and 12th days of March, A. D. 1886.

HERMANN GRÜNEBERG.

EMIL BLUM.

Witnesses as to the signature of Hermann Grüneberg:

E. WEYRACK,

JOS: RAWE.

Witnesses as to the signature of Emil Blum:

B. ROY,

M. W. MOORE.