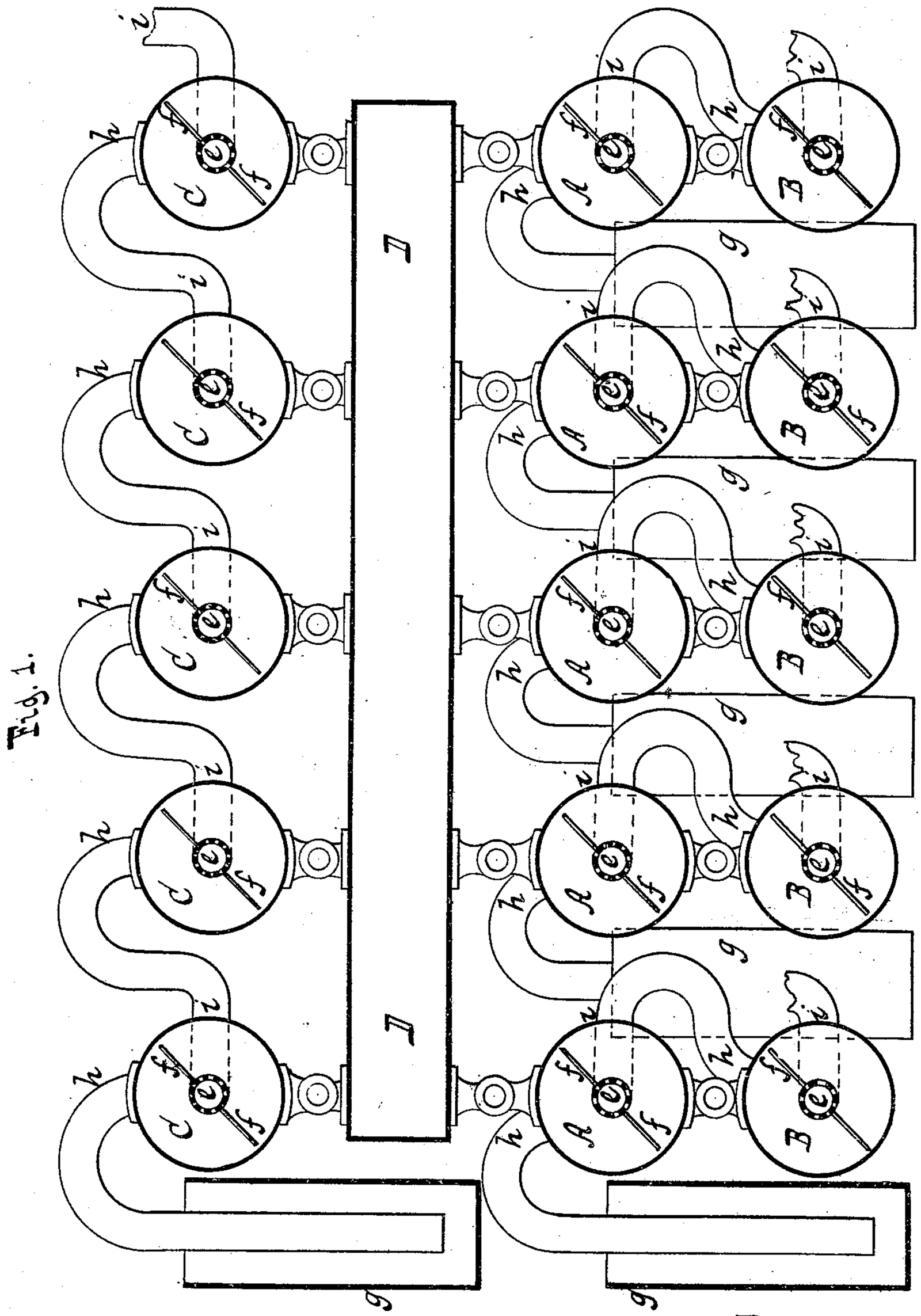
(No Model.) 2 Sheets—Sheet 1.

H. J. E. HENNEBUTTE.

APPARATUS FOR MANUFACTURING SODA BY THE AMMONIA PROCESS.

No. 262,767. Patented Aug. 15, 1882.



Witnesses
William Miller
Otto Hufeland

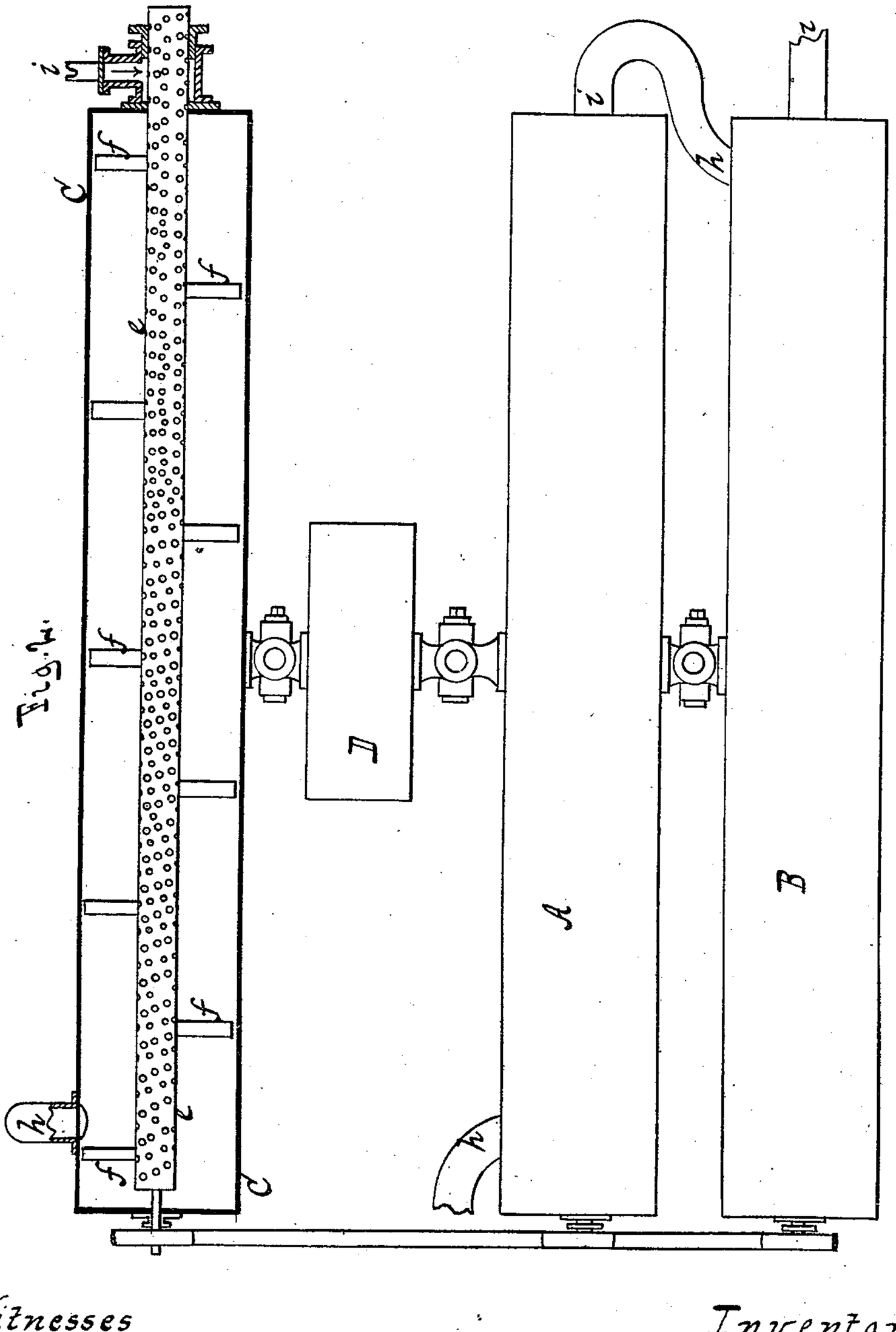
Inventor
Henri Joseph Ernest Hennebutte
by Van Santwood & Hauff
his att'ys

(No Model.) 2 Sheets—Sheet 2.

H. J. E. HENNEBUTTE.

APPARATUS FOR MANUFACTURING SODA BY THE AMMONIA PROCESS.

No. 262,767. Patented Aug. 15, 1882.



Witnesses
William Miller
Otto Hupfeld

Inventor
Henri Joseph Ernest Hennebutte
by Van Santvoord & Hauff
his attys

UNITED STATES PATENT OFFICE.

HENRI J. E. HENNEBUTTE, OF ANGLET, ASSIGNOR TO THE SOCIÉTÉ ANONYME DES PRODUITS CHIMIQUES DU SUD-OUEST, OF PARIS, FRANCE.

APPARATUS FOR MANUFACTURING SODA BY THE AMMONIA PROCESS.

SPECIFICATION forming part of Letters Patent No. 262,767, dated August 15, 1882.

Application filed December 23, 1881. (No model.) Patented in France June 15, 1881, No. 142,090.

To all whom it may concern:

Be it known that I, HENRI JOSEPH ERNEST HENNEBUTTE, a citizen of France, residing at Anglet, in the Department of the Basses-Pyrénées and Republic of France, have invented new and useful Improvements in Apparatus for the Manufacture of Soda, of which the following is a specification.

The object of the present invention is to provide an efficient apparatus for manufacturing soda by the ammonia process; and to such end the invention consists in the novel construction and combination of mechanism illustrated in the accompanying drawings, in which—

Figure 1 represents a vertical sectional view of the apparatus embodying my invention; and Fig. 2, a side view, partly in section.

I will first describe the mode of operation of the apparatus in manufacturing soda by the ammonia process.

First operation: Into a battery of horizontal cylinders, of sufficiently small diameter to allow them to be subjected to intense refrigeration, is introduced a thoroughly saturated solution of chloride of sodium, care being taken that each cylinder be not more than about two-thirds filled. Each cylinder carries in the center a hollow shaft perforated with holes serving for the introduction of the gases into the liquid. The gases, however, may be introduced into the cylinder by any other method, provided that the gases are divided or distributed in the liquid, and that the tubes forming a communication have their mouths in the chamber or empty space above the liquid and terminate in the body of the liquid in the succeeding cylinder. The hollow movable shaft carries also a rake or stirrers, the object of which is to stir up at certain periods of the operation the precipitates which have formed in the liquor. The gases which are freed in the first of the cylinders C pass up through a communicating tube rising from the upper part of the same, and which fits to the hollow shaft of the next adjacent cylinder C, and by the same means the different cylinders composing a battery communicate with each other. If, by means of the hollow shaft of the first cylinder of the series C, ammoniacal gas supplied from generators is introduced, the ammonia and carbonate of ammonia set free partly saturate first the contents of the first cylinder of the series C to ascertain extent, then those of the adjacent cylinder of the series C, and the operation is continued until the whole mass of the liquid contained in the cylinders composing the battery contains on an average ten per cent. of ammonia, (NH_3.) At this stage of the operation each of the cylinders C contains a varying richness of ammonia—that is to say, the first is saturated, while the others have some traces less of ammonia.

Second operation: This consists in replacing in the first cylinder of the series C the current of ammoniacal gases by a strong current of impure carbonic acid coming from lime-furnaces. This part of the operation is only ended when it is estimated that the point of simple carbonation has been somewhat passed—that is to say, when a formation of sesquicarbonate begins. This operation, as the following ones, is carried on without pressure. A simple water-valve suffices for a satisfactory utilization of the gases, and experience has proven that a maximum layer of one meter of water hindered appreciable losses of carbonic acid. Under the conditions above described the gaseous (impure carbonic acid) current acts as follows: Entering into a very rich ammoniacal solution, it takes up a considerable quantity of extremely-hydrated ammonia, with which it unites in the form of gas in the upper part of the chamber of the cylinder. A part of the carbonate of ammonia formed falls back into the liquid, where it is dissolved, while the excess passes into the following cylinders.

Third operation: Each one of the cylinders of the battery which has been described carries a waste-pipe, all of equal cross-section, and each one of these tubes leads into a receiver or horizontal cylinder, D, this arrangement allowing, with a varying richness in each cylinder, of obtaining liquors possessing an average composition. The ammoniacal brine, obtained as aforesaid, passes from the receiver D into a series of small batteries, each composed of two cylinders, in all respects like the preceding, with this difference that the cylinder B of this new battery is placed below the cylinder A. The upper cylinder, A, of this

second battery is filled about three-fourths, and the lower cylinder, B, is left entirely empty. The cylinder A is provided with a water-valve, with a mean height of water of about one meter. Then it is only necessary to introduce pure carbonic acid, coming from the calcination of bicarbonate of soda, into the cylinder B, which, as already stated, is empty. This pure carbonic acid immediately rises into the cylinder A, where it is readily absorbed, while the liquids contain only sesquicarbonate, and even a small quantity of bicarbonate. The operation is known to be finished by the water-valve no longer sufficing to retain the gas. In the cylinder A the operation takes place to a great extent as in the previously-described stage—that is to say, the carbonic acid which passes into the small layer of liquid attacks the carbonate of ammonia, with which it unites in the form of gas in order to fall back into the liquid and dissolve there.

Fourth operation: The liquids contained in the cylinder A, and which contain principally sesquicarbonate of soda, are led off into the cylinder B in order to be there bicarbonated by the pure carbonic acid obtained from the calcination of the bicarbonate of soda. This carbonic acid passes in a strong current through the above-named liquid, and is finally condensed in the upper cylinder, A. The bicarbonation is thus obtained by an excess of gas, which excess is utilized, moreover, in the upper cylinder. The bicarbonate of soda which constitutes the product of this operation is turbinated or passed through a filter-press in order to separate therefrom the ammoniacal mother-liquors.

Fifth operation: The regeneration of the ammonia contained in the mother-liquors is obtained by means of the distilling apparatuses of Hennebutte and Vauréal.

As is seen by the foregoing remarks, in the second operation the impure gases coming from the lime-furnace are deprived of their carbonic acid by their passage in the cylinders, and it is chiefly in a gaseous state that this carbonic acid is combined with the extremely-hydrated ammonia violently acted on by the volume of gas.

In the third operation the pure carbonic acid coming from the furnaces for calcining soda acts on a part of the carbonate of ammonia in the chamber of the cylinder A, with which it also combines in a gaseous form.

Finally, in the fourth operation the bicarbonation of the liquids contained in the cylinder B is obtained by a superabundant quantity of pure carbonic acid, of which the excess is entirely utilized for the use of the cylinder A.

I will now proceed to give a detailed description, by reference to the drawings, of the parts before referred to.

The cylinders composing the first series are indicated by the letter C. As is seen, these cylinders are provided with inlet-tubes *e*, hung in bearings, and to which revolving motion may be imparted by belts and pulleys operated by suitable motive power. The inlet-tubes *e* are perforated to allow the gas to flow into the cylinders C. These tubes *e* are also provided with stirrers *f* for stirring up the liquid, said stirrers *f* being actuated by the revolving tube *e*. The gas is led into the tube *e* through a tube, *i*, which in the case of the first cylinder leads from the generator, and in the case of the succeeding cylinders connects with a curved arm or tube, *h*, leading from the upper part of the next preceding cylinder. In the case of the last cylinder C, the tube *h* leads into a water-valve formed by submerging the outlet of said tube in water contained in the tank *g*.

The cylinders A and B correspond in their construction to the cylinders C, just described, and the communicating tubes *h i* and inlet-tubes *e* in these cylinders A and B, as also the water-valve connected therewith, are similar in construction and operation to those described in connection with the cylinders C, and are arranged as shown. The tubes *e* in the cylinders A and B also carry stirrers *f*.

The communication between the cylinders C, receiver D, and cylinders A and B for the passage of the liquid is formed by stop-cocks, as shown in the drawings. The gas passes into the hollow perforated inlet-tubes *e* by means of a tubular passage in the stuffing-box at the side of the cylinder C. The communicating tubes *h* may be simply bolted onto the cylinders C. The different carbonating-cylinders are independent of one another, the tubes and faucets connecting them being bolted, which allows the battery to be readily taken apart. To secure air-tight joints washers or leaden rings are employed.

The cylinders C receive the brine through a tube provided with a faucet, (not shown in the drawings,) which may be placed at any convenient place along the cylinder. Sheet-iron is most preferable for the construction of the cylinders. Metals other than iron cannot be used. It is of course understood that the bearings in which the perforated tubes *e* revolve are made air-tight by means of stuffing-boxes.

As has been mentioned, the cylinders are of sufficiently small diameter to be subjected to intense refrigeration, which refrigeration may be accomplished by causing to circulate about the cylinders any refrigerating-liquid. The diameter of each of the cylinders composing a carbonating apparatus varies from 0.60 to 0.70 meter. The point of saturation by the carbonic acid or degree of carbonation is estimated by the volumetric analysis of the carbonic gas caused to unite with the bases. The carbonation of the brine takes about six hours, the sesquicarbonation about six hours, and the bicarbonation about six hours. The formula representing the reaction producing soda may be stated as follows: $NaCl+NH_3+H_2O+CO_2=CO_3NaH+NH_4Cl$.

The regeneration of the ammonia contained

in the mother-liquors can be obtained by means of apparatus for distilling ammoniacal liquors, such as described in a specification pertaining to another application for a United States patent for apparatus for distilling ammoniacal liquors signed by me and de Vauréal June 2, 1881.

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

1. The herein-described apparatus, consisting of primary cylinders, C, secondary cylinders, A B, and receiver D, communicating with said cylinders, said cylinders being provided with inlet-tubes *e* and rakes or stirrers *f*, substantially as set forth.

2. The herein-described apparatus, consisting of primary cylinders, C, secondary cylinders, A B, and receiver D, communicating with said cylinders, said cylinders being provided with inlet-tubes *e*, rakes or stirrers *f*, and water-valves *g*, substantially as set forth.

3. The herein-described apparatus, consisting of primary cylinders, C, and secondary cylinders A B, and receiver D, communicating with said cylinders, said cylinders being provided with inlet and communicating tubes *e h i* and rakes or stirrers *f*, substantially as set forth.

4. The herein-described apparatus, consisting of primary cylinders, C, and secondary cylinders, A B, and receiver D, communicating with said cylinders, said cylinders being provided with inlet and communicating tubes *e h i*, rakes or stirrers *f*, and water-valves *g*, substantially as set forth.

5. The herein-described apparatus, consisting of primary cylinders, C, and secondary cylinders, A B, said cylinders being provided with perforated revolving tubes *e* and rakes or stirrers *f*, supported on said tubes, substantially as and for the purpose set forth.

6. The herein-described apparatus, consisting of primary cylinders, C, and secondary cylinders, A B, with the connecting-pipes, said cylinders being provided with tubes *e*, perforated longitudinally and extending throughout the length of such cylinder, substantially as set forth.

In testimony whereof I have hereunto set my hand and seal in the presence of two subscribing witnesses.

HENRI JOSEPH ERNEST HENNEBUTTE. [L. S.]

Witnesses:

RENÉ FRANCK,

ELYSÉE LÉON.