

Waltz & Pendleton,
Making Carbonate Soda.
No. 90,140. *Patented May 18, 1869.*

Fig. 1.

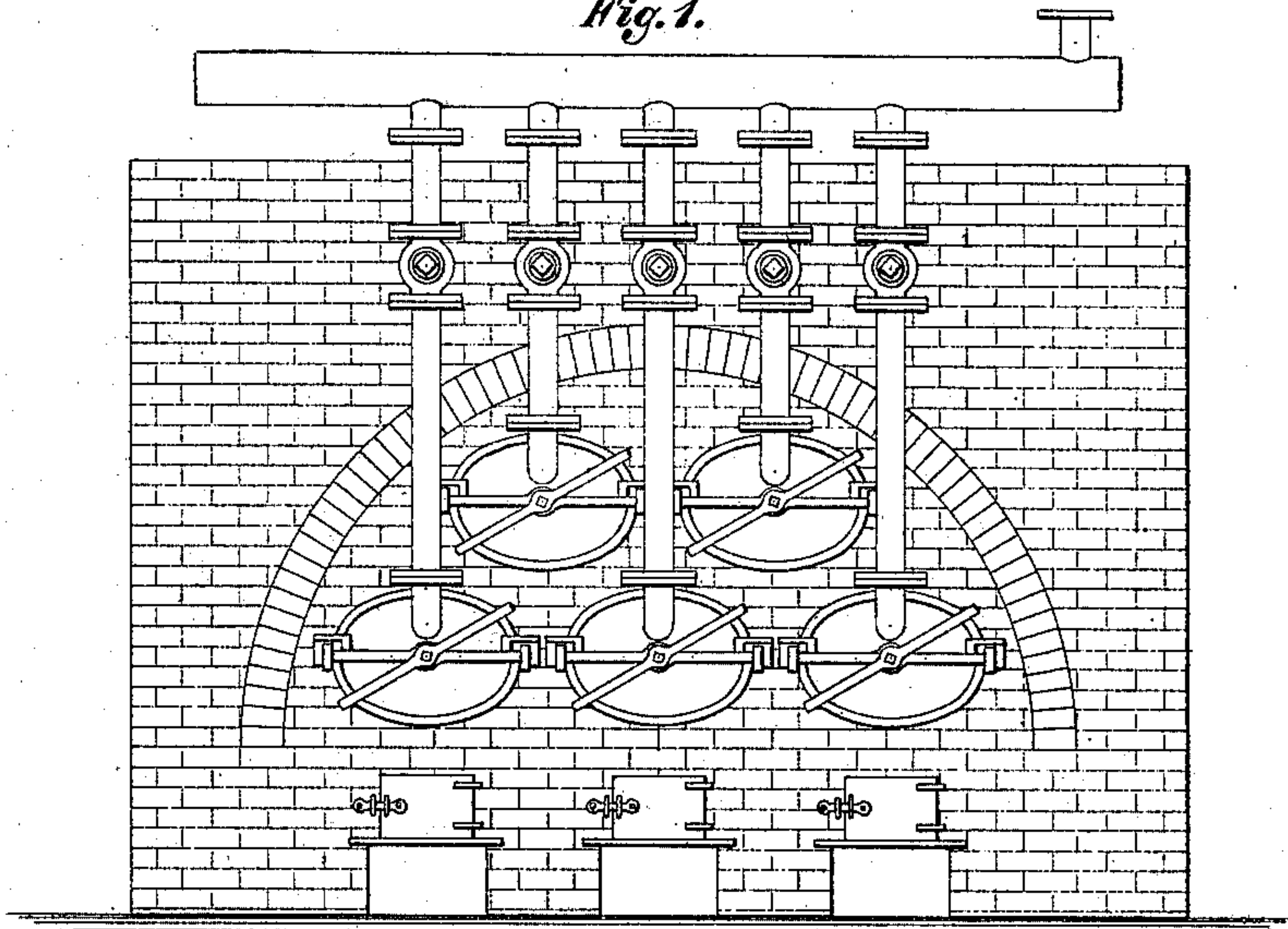
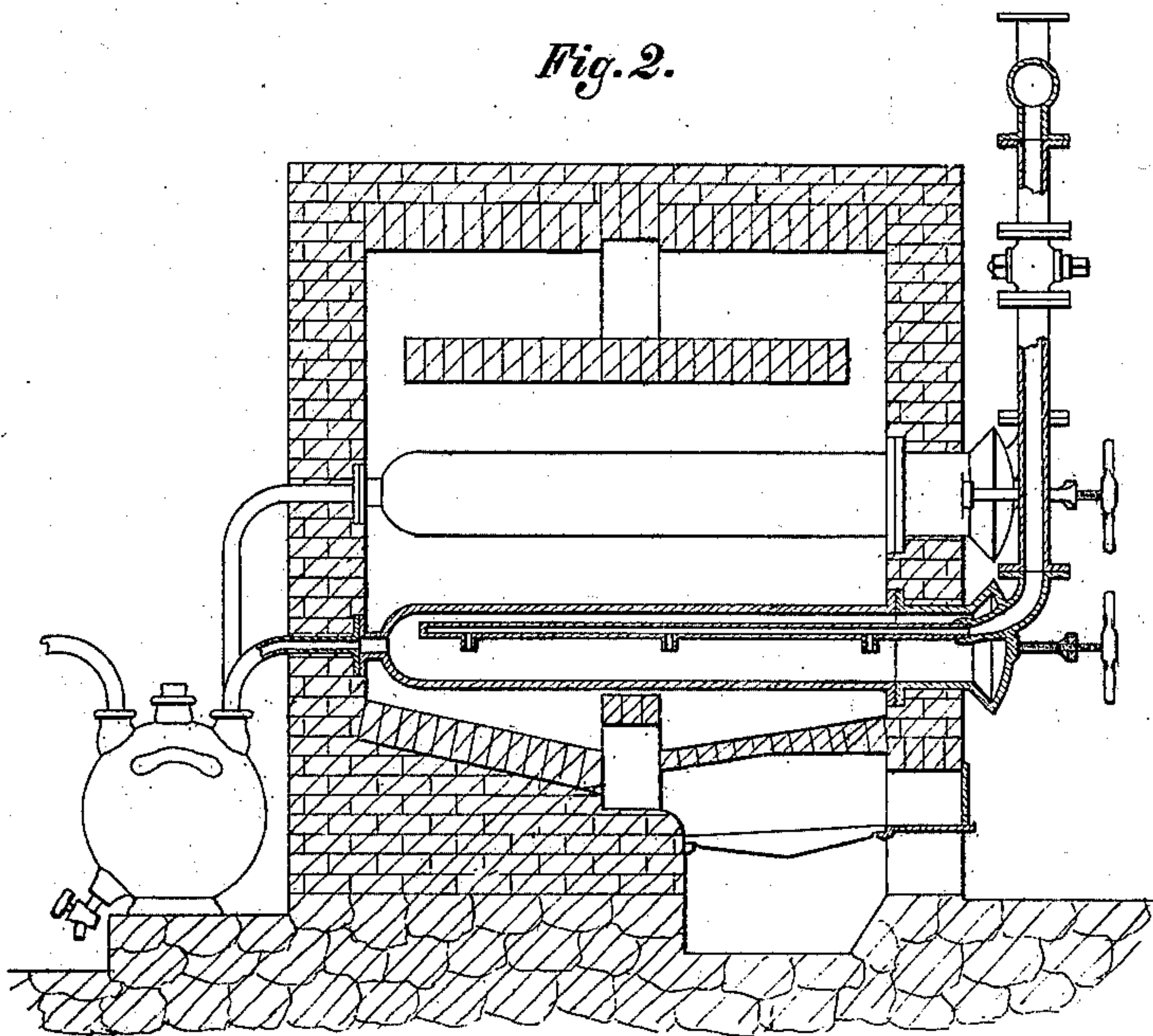


Fig. 2.



Witnesses.

W. C. Livingston

Inventors

*Walter Waltz, by their attorneys,
 John M. Pendleton.*

United States Patent Office.

ISIDOR WALZ AND JOHN M. PENDLETON, OF NEW YORK, N. Y.

Letters Patent No. 90,140, dated May 18, 1869.

IMPROVEMENT IN THE MANUFACTURE OF CARBONATE OF SODA AND OTHER CHEMICALS.

The Schedule referred to in these Letters Patent and making part of the same.

To all whom it may concern:

Be it known that we, ISIDOR WALZ and JOHN M. PENDLETON, both of the city and county of New York, State of New York, have invented a new and useful Method for the Manufacture of Carbonate of Soda and other Chemicals; and we do hereby declare that the following is a full and exact description thereof.

We will first describe what we consider the best means of carrying out our invention, and will afterwards designate the points which we believe to be new therein.

We take carbonate of lime, either as obtained artificially or as it occurs in nature, and nitrate of soda, either as artificially prepared, or as it occurs in nature, both preferably in a dry, pure, and pulverulent condition, and mix well together, in the proportion of seventeen parts of nitrate of soda to ten parts of carbonate of lime, or in such larger proportions of carbonate of lime as may be deemed expedient, and fill a cylindrical retort to from one-third part to three-fourths parts of its capacity in a horizontal direction. This retort is then placed in a furnace, constructed mainly like the furnaces used for heating the retorts in the manufacture of illuminating-gas. At its rear end each retort terminates in a tube which passes through and projects beyond the rear wall of the furnace. The front end of such retort rests in the front wall of the furnace, so that the retort shall have a horizontal position. Each retort should be provided with a cover, which may, at will, be taken therefrom or connected therewith firmly and tightly. Such cover has an opening for the introduction of a pipe, which serves the purpose of introducing steam into the retort. It extends from the front of the retort to within a few inches of its rear end, and is provided with one or more openings suitably disposed within the retort. We then connect that part of this pipe, which projects beyond the retort in front by a proper coupling, with an iron tube connecting with a steam-boiler, and provided with proper cocks, valves, or other contrivances for admitting, regulating, or shutting off the steam. We connect the rear end of the tube which projects beyond the rear wall of the furnace, by proper joints and couplings with a series of large stone-ware-receptacles, known as Woulfe's bottles, which shall contain a larger or smaller amount of water. The furnace contains from one to five retorts, pipes, covers, tubes, and connections, as above described. We then heat the furnace with coal or coke, or other carbonaceous material, so that the retorts and their contents shall attain a temperature from dark to bright red. We at the same time admit steam into the retort by means of the appliances above described. At such temperature the chemical and physical changes, combinations, and decompositions taking place in the retort may be expressed by the following chemical formula: $\text{CaOCO}_2 + \text{NaONO}_2 + \text{HO} = \text{CaO} + \text{NaOCO}_2 + \text{N}_2\text{O}_5 + \text{HO}$, which may be interpreted as follows:

First, the nitrate of soda is decomposed into anhy-

drous oxide of sodium on the one hand, and nitric acid on the other hand.

Second, the nitric acid is decomposed by the heat into oxygen and combinations of nitrogen, with less oxygen than is contained in nitric acid.

Third, the carbonate of lime is decomposed into anhydrous oxide of calcium (or quick-lime) on the one hand, and carbonic acid on the other hand.

Fourth, the anhydrous oxide of sodium (or caustic soda) unites chemically with the carbonic acid, and forms mainly, or altogether, carbonate of soda.

Fifth, the steam introduced into the retort becomes superheated in contact with the heated mass and the heated sides of the retort.

Sixth, this superheated steam reunites the oxygen with the lower combinations of nitrogen and oxygen, so as to regenerate nitric acid, and unites therewith to form a gaseous solution of this acid.

The final products of this process are—

First, a gaseous solution of nitric and hyponitric acids in steam. This is carried through the tube forming the rear end of the retort into the above-mentioned series of Woulfe's bottles, and condensed therein, forming more or less concentrated solutions of nitric and nitrous acids. These solutions are distilled and concentrated together, or separately, according to the methods in existence for this purpose, so as to produce more or less concentrated solutions of commercial or pure nitric acid.

Second, a sintered or melted mass, consisting of quick-lime and carbonate of soda, and possibly of some caustic soda. The fire is extinguished, the retorts allowed to cool, and this mass taken out and suitably broken up.

(A.) This mass may now be brought directly into the market, and may be used to produce solutions of caustic soda by dividing it into small fragments, and placing them in vats, tubs, or other receptacles, with sufficient water, which may be heated by steam, or directly by fire, when the following change will take place: $\text{NaOCO}_2 + \text{CaO} + \text{HO} = \text{CaOCO}_2 + \text{NaOHO}$; that is to say, the water will dissolve the carbonate of soda, and leave the lime undissolved. The higher temperature will aid this dissolving-process, and cause the formation of carbonate of lime and a solution of caustic soda.

(B.) Or by another method of operating, we take this mass, suitably comminuted, and place the same in a vat with a sufficient quantity of water to dissolve all the carbonate of soda. The process of dissolving may be aided and accelerated by stirring or otherwise agitating the contents of said receptacle. The lime is allowed to settle and the supernatant liquid, which forms a more or less concentrated solution of carbonate of soda, drawn off and concentrated, evaporated or crystallized, according to the usual methods employed for this purpose.

(C.) Or, again, if preferred, we can take this mass suitably comminuted and place the same with water

(sufficient to dissolve all of the carbonate of soda present) in a vat which must be provided with appliances for raising the temperature. We then raise the temperature to the boiling-point when the following chemical change will take place: $\text{NaOCO}_2 + \text{CaO} + \text{HO} = \text{NaOHO} + \text{CaOCO}_2$, which may be interpreted as follows:

Carbonate of lime is formed which is insoluble in the supernatant liquid, and sinks to the bottom, and a more or less concentrated solution of caustic soda is formed, which is drawn off, concentrated, evaporated, and reduced to a dry state by the usual methods employed for that purpose.

The carbonate of lime formed is dried, and may be employed to form a fresh mixture with nitrate of soda, as above described.

We can use in our process carbonate of lime in the form of limestone, marble, calspar, or any other mineral containing lime and carbonic acid in a dry or moist, pure or impure, crystalline or amorphous, or pulverulent condition, and nitrate of soda in the form of Chili nitre, or Chili saltpetre, or soda saltpetre, or any other mineral (by whatever name it may be known,) containing soda and nitric acid in a dry or moist, pure or impure, crystalline or amorphous, solid or pulverulent condition; but we prefer both in a dry and finely-divided condition, as above stated.

We can use all ordinary materials for the vats or tubs for the final treatment, and heat by any convenient means; but we prefer hard wood for the vessels and heating by steam-pipes when plenty of steam can be conveniently commanded.

The accompanying drawings represent the apparatus we employ to carry out our invention.

Figure 1 is a front view, and

Figure 2, a vertical section of the apparatus.

We are aware that Karl Lieber has, in his patent dated December 15, 1868, described certain improvements. Such we do not claim. Neither do we claim as new anything in the above connected with the form, material, or arrangement of the various furnaces, retorts, vats, tubs, or other receptacles and Woulfe's bottles, or anything connected with the working up the first solutions of nitric acid as condensed in the Woulfe's bottles, or with the working up of the mass, consisting of quick-lime and carbonate of soda, into a marketable state; but

We claim, in the process of manufacture above described—

1. The use of steam in connection with a heated mass of carbonate of lime and nitrate of soda in a highly-heated vessel or retort for the regeneration of nitric acid, substantially as above described.

2. The production of a mass, consisting of quick-lime and carbonate of soda, in exactly or nearly their chemical proportions or equivalents, and forming a mass applicable to the production of caustic-soda lyes from carbonate of lime and nitrate of soda, treated in the manner substantially as herein described.

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Witnesses:

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