(No Model.)

T. CRANEY.

PROCESS OF AND APPARATUS FOR MAKING CARBONATES OF SODA.

No. 552,895. Patented Jan. 14, 1896.

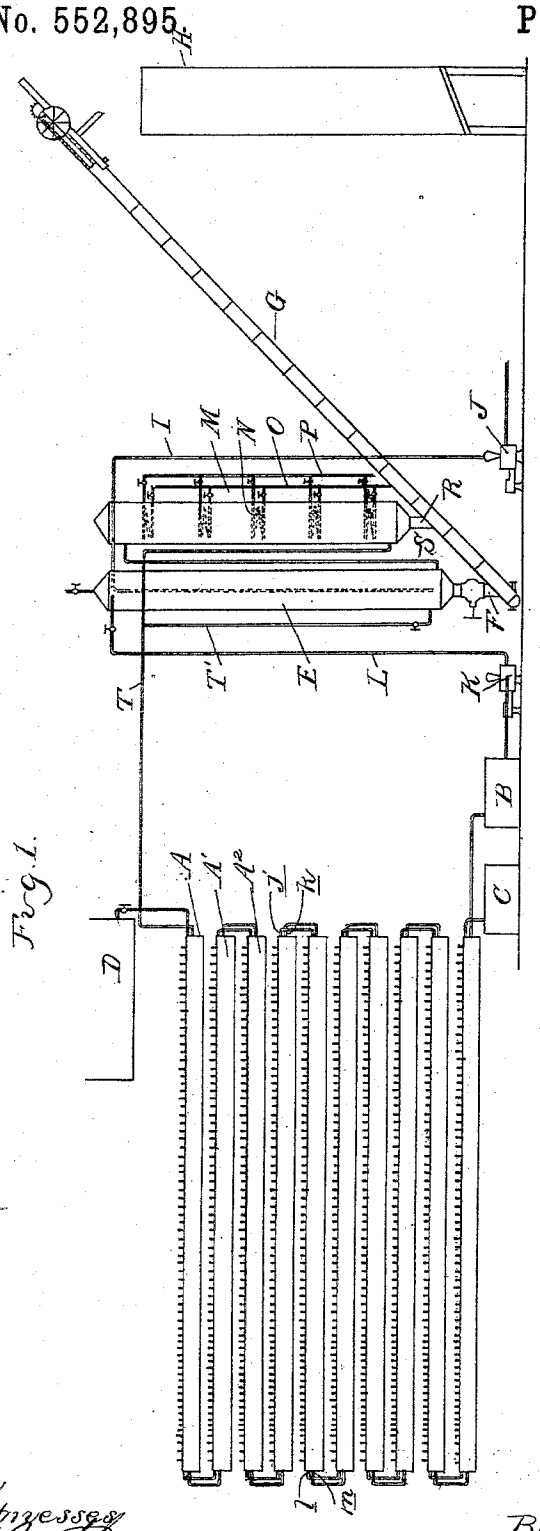

Fig. 1.

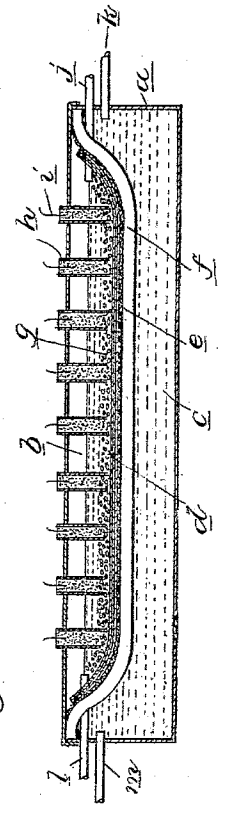

Fig. 2.

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UNITED STATES PATENT OFFICE.

THOMAS CRANEY, OF BAY CITY, MICHIGAN.

PROCESS OF AND APPARATUS FOR MAKING CARBONATES OF SODA.

SPECIFICATION forming part of Letters Patent No. 552,895, dated January 14, 1896.

Application filed May 15, 1894. Serial No. 511,371. (No model.)

To all whom it may concern:

Be it known that I, THOMAS CRANEY, a citizen of the United States, residing at Bay City, in the county of Bay and State of Michigan, have invented certain new and useful Improvements in the Process of and Apparatus for Making Carbonates of Soda, of which the following is a specification, reference being had therein to the accompanying drawings.

The object of this invention is to make mono and bi carbonate soda on a commercial scale by the electrolysis of salt brine; and to this end my invention consists in the electrochemical process and apparatus hereinafter described.

In the drawings, Figure 1 is a diagrammatic elevation of an apparatus devised for carrying out my process. Fig. 2 is a vertical central cross-section of one of the electrolytic vats.

A A' A^2 represent a number of electrolytic vats arranged in vertical series. These vats may be of any of the known constructions in which there is a diaphragm dividing it into anode and cathode compartments. The electrolytic vat shown in the drawings is of the character shown and described by me heretofore in Letters Patent numbered 520,257, and dated May 22, 1894. It is adapted for this apparatus on account of its convenient form which allows of the use of a large number in vertical series in a limited space and also on account of its permanent character. Briefly described, its construction is as follows: The outer tank *a*, which is of boiler-iron, is divided into an anode-compartment *b* and a cathode-compartment *c* by an electrolytic diaphragm *d*, which is preferably formed of several thicknesses of asbestos paper. This diaphragm rests upon a metal screen *e*, which in turn is supported upon iron girders *f* secured in the outer tank. Upon the diaphragm is placed a layer of carbon *g*, which forms the anode, with which electrical connection is made through a number of carbon pencils *h*. These carbon pencils are connected by wire terminals *i* with the positive pole of the source of electricity, and for greater permanency they are inclosed in open-ended tubes of vitrified stoneware. The negative terminal is connected to the metallic supporting-frame of the diaphragm, which thereby forms the cathode. Each vat is provided with separate feed-pipes *j k* into its anode and cathode compartments, and separate overflow discharge-pipes *l m* from said compartments respectively, and in connecting the vats in series, as shown, the discharge-pipes from the topmost vat are connected to the corresponding feed-pipes of the vat next below, and so on through the entire series.

B is a receiver into which the discharge overflow from the cathode-compartment of the last vat communicates. C is a like receiver into which the discharge overflow from the anode-compartment of the last vat communicates.

D is a source of supply of salt brine which communicates into the anode-compartment of the first vat through a valved supply-pipe.

E is a carbonator having the character of a large stand-pipe provided at its lower end with a valve-controlled discharge-outlet F, through which it connects into the inclined trunk of a conveyer G, which is adapted to discharge into a draining-bin H.

I is a gas-supply pipe leading from a pump J, which is connected to a suitable source of supply of carbonic-acid gas (not shown) into the carbonator and extending down interiorly to near the bottom thereof.

K is another pump for pumping the liquid from the receiver B through a valve-controlled supply-pipe L into the top of the carbonator.

M is a cooling-vessel similar in character to the carbonator E, and containing one or more coils N, through which a refrigerating liquid can be conducted by means of the pipes O P, which connect the coils in circuit with a refrigerating source. (Not shown.)

R is a discharge-outlet connecting the bottom of the cooling-vessel with the trunk of the conveyer.

S is a pipe leading from near the bottom of the carbonator into the top of the cooling-vessel.

T is a pipe leading from the bottom of the cooling-vessel compartment of the topmost electrolytic vat.

The parts being thus constructed and arranged, as shown and described, the apparatus is intended to carry out the following process: The electrolytic vats are organized for

the continuous production of the caustic soda in solution from salt brine. To this end salt brine from any suitable source of supply is fed into the anode-compartment of the top vat, preferably in a continuous manner. By overflowing from one anode-compartment into the one next below all the vats are kept continuously supplied and the overflow from the last vat flows into the receiver C. During its flow through the vats electrolytic action takes place and a portion of the salt is decomposed, producing chlorine and caustic soda. The chlorine passes off with the solution of the undecomposed salt brine into the receiver C and is recovered therefrom for the manufacture of other valuable by-products, while the salt brine may be used over again. The caustic soda is transferred by the electrolytic action into the cathode-compartments, which together with all the parts in communication therewith—such as the receiver B, carbonator E, cooling-vessel M, and trunk G of the conveyer—may at the start be filled with fresh water alone. If now the pump K is set into action, liquid is transferred from the receiver B through pipe L into the carbonator E, from there by displacement through pipe S into the cooler M, and through pipe T from the cooler into the cathode-chamber of the top vat, from which it overflows into the cathode-compartment of the vat below, and successively through the other like compartments, and finally from the last one into the receiver B. In this manner the liquid with which the apparatus is charged on the soda side is constantly circulated during the entire process, and thus it receives while passing through the cathode-compartments of the vats a constant increment of caustic soda, which, as the quantity of the liquid is fixed, increases the strength of the solution. Now having a constant supply of carbonic-acid gas (which may be produced in the usual manner as a product of combustion of carbon) I connect the pump J with this supply and inject the gas through the pipe I into the carbonator. If the intention is to make monocarbonate of soda, the quantity of carbonic-acid gas is regulated to the amount required to transform the solution of caustic soda into a monocarbonate, which being very soluble at ordinary temperatures remains in solution and passes from the carbonator through the pipe S into the cooling-vessel, wherein it is now subjected to a reduction of temperature by passing a suitable refrigerating-liquid through the pipes O P.

As the solubility of monocarbonate of soda is very much less at a low temperature (it being about one hundred and fifty at 77°, while at 50° it is only forty-one parts of its weight in one hundred parts of water) it is obvious that after the monocarbonate in solution has attained a certain strength a portion of it will be precipitated and fall to the bottom of the cooler, and thence through the discharge-outlet into the trunk G, from which it is removed by the conveyer and discharged into the bin H, which is properly constructed to drain the crystal from the adhering solution. A portion of monocarbonate of soda will remain in solution and pass from the cooler through the pipe T back into the electrolytic vats, where its strength is continually increased by the addition of fresh caustic soda during its passage through the cathode-compartments, while its temperature will be raised again by the heat produced as an accompaniment of the electrolytic action. In this manner monocarbonate of soda will be continually precipitated in the cooler by the constant increment of caustic soda derived from the passage of the liquid through the electrolytic vats.

The water contained on the soda side of the apparatus—that is, in the cathode-compartments and in the parts connected in circulation with it—is fixed in quantity and acts merely as a carrier. A constant loss of it, however, occurs by evaporation and by its mechanical and chemical combination with the solid product of monocarbonate removed from the apparatus. Fresh water may be added from time to time, as required, to keep up the circulation.

From the operation it will be seen that instead of using fresh water from the start to charge the soda side of the apparatus a solution of caustic soda or monocarbonate of soda may be used, which would hasten the production of the precipitate. It is even permissible to use salt water as the vehicle on the soda side, provided the small amount of salt with which the product would then be liable to be contaminated would cause no objection.

The connection of the carbonator with the trunk G of the conveyer is for the purpose of removing any crystals of monocarbonate or other deposit which may collect on the bottom, as by opening the valve in said connection the crystals or other deposit may be discharged into the conveyer-trunk. Any tendency to the formation of crystals in the carbonator may, however, be avoided by placing a heating-coil into the carbonator so as to raise the temperature sufficiently to hold a larger amount of monocarbonate in solution.

For obtaining bicarbonate of soda as a product the process is in so far modified that an increased quantity of carbonic-acid gas is conducted into the carbonator, enough to transform all the caustic soda or monocarbonate into bicarbonate of soda, and as this is less soluble than the monocarbonate a portion of it will be precipitated in the carbonator and collect at the bottom, from which it can be from time to time discharged into the conveyer. The bicarbonate remaining in solution can be returned either directly to the vats through a suitable connection T′, which forms a by-pass into the pipe T, or it may be conducted into the cooler M, and by lowering the temperature of the solution an additional precipitation of bicarbonate may be obtained. By conducting the process in this manner a

solution of bicarbonate is returned to the electrolytic vats, and by the addition of caustic soda this is transformed in the vats into monocarbonate and returned as such to the carbonator, to be again transformed into bicarbonate. This process of feeding a solution of bicarbonate into the soda side of the electrolytic vats materially assists its electrolytic action on account of the chemical affinity of the caustic soda and the bicarbonate of soda.

In constructing my apparatus for carrying out this process I adopt the required relative proportions between the parts to adapt the apparatus to operate in its different parts as a continuously-operating plant in the manner described.

What I claim as my invention is—

1. The method of making carbonate of soda, consisting in continuously circulating water containing the carbonate to be obtained in saturated solution through the cathode compartment of an electrolytic apparatus, in adding continuously caustic soda to the solution while passing it through the cathode compartment by the electrolysis of salt brine supplied to the anode compartment, in introducing into the solution after its passage through the cathode compartment carbonic acid gas as required to convert all the solution into a solution of the carbonate to be obtained, in precipitating from said solution the increment of carbonate thus produced and removing the same, and in conducting the remaining solution back again into the cathode compartment, substantially as described.

2. The process of making mono-carbonate of soda, which consists in circulating water containing mono-carbonate of soda in saturated solution through the cathode compartment of an electrolytic apparatus, in continuously adding caustic soda to the solution while passing it through the cathode compartment by the electrolysis of salt brine continuously supplied to the anode compartment, in introducing into the solution after its passage through the cathode compartment carbonic acid gas as required to convert the free soda into a mono-carbonate, in cooling the solution after its treatment with carbonic acid gas and thereby precipitating mono-carbonate crystals, in removing the crystallized monocarbonate from the solution and in supplying the remaining solution to the cathode compartment of the electrolytic apparatus, substantially as described.

3. In an apparatus for manufacturing monocarbonate of soda, the combination of an electrolytic apparatus having separate feed and discharge pipes for its anode and cathode compartment, a closed circuit between the feed and discharge pipes of the cathode compartment, a pump in said circuit for maintaining a continuous circulation in said closed circuit, a carbonator and a cooler included in said circuit and provided with valved discharge openings therefrom, and a by-pass around the cooler, substantially as described.

4. In an apparatus for manufacturing soda, the combination of a vertical series of electrolytic vats organized for the continuous production of caustic soda from salt brine and provided with feed and discharge connections for the continuous flow of the product through the compartments, a receiver B, the carbonator E, a cooler M, intermediate connections connecting the cathode compartments of the electrolytic vats in circuit with said receiver, carbonator and cooler, a pump for producing a continuous flow of the liquid through said circuit, a conveyer provided with an inclined trunk G and the discharge connections F R, from the bottom of the carbonator and cooler into the trunk of the conveyer, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

THOMAS CRANEY.

Witnesses:
M. B. O'DOGHERTY,
O. F. BARTHEL.