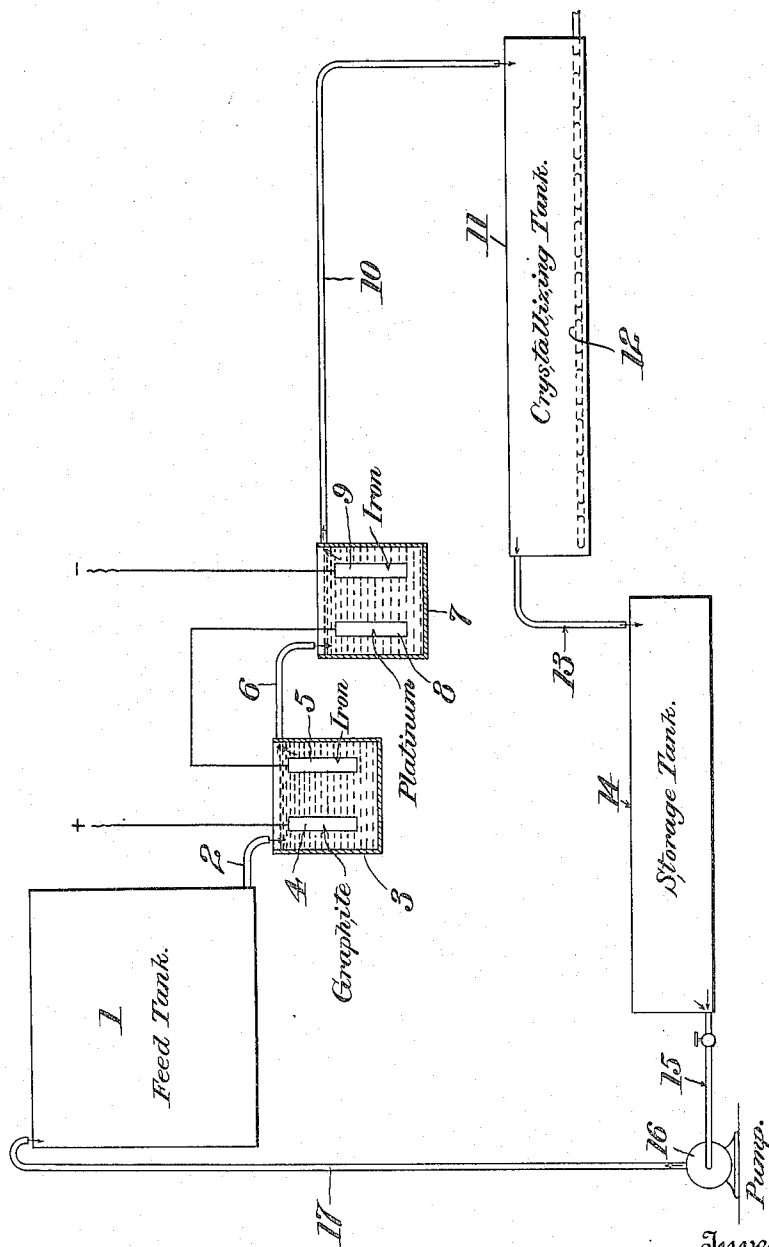


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 PROCESS OF PRODUCING CHLORATES OF ALKALIS AND ALKALINE EARTHS.  
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1,143,586.

Patented June 15, 1915.



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# UNITED STATES PATENT OFFICE.

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## PROCESS OF PRODUCING CHLORATES OF ALKALIS AND ALKALINE EARTHS.

1,143,586.

Specification of Letters Patent.

Patented June 15, 1915.

Application filed September 12, 1914. Serial No. 861,395.

*To all whom it may concern:*

Be it known that I, WALTER LAIB, a citizen of the United States, residing at Rittman, in the county of Wayne and State of Ohio, have invented a certain new and useful Improvement in Processes of Producing Chlorates of Alkalis and Alkaline Earths, of which the following is a full, clear, and exact description.

15 The object of this invention is to provide for the manufacture of chlorates of alkalis and alkaline earths by electrolysis, and more particularly the manufacture of potassium chlorate.

20 Prior to this invention, electrolysis, in connection with the application of extraneous heat as a subsequent step in conversion, has been used in the production of chlorates, but in the present invention electrolysis alone is used.

While the invention may be utilized in the production of various chlorates, I will describe it as applied to the production of potassium chlorate.

25 The invention consists in exposing a solution of potassium chlorid to a preliminary electrolysis, at a relatively low temperature, in electrolytic cell having a graphite or other carbon anode, and a suitable durable cathode, such as iron, and then further electrolyzing the thus electrolyzed solution in another electrolytic cell having a platinum or equivalent permanent anode, and an iron or other durable cathode, in the presence of a higher temperature due to the electric current, and then conveying the electrolyzed solution to a cool crystallizing tank, from which the crystals are removed from time to time, and the liquor run thence into a storage tank, where it is refreshed or resaturated, whence it may be conveyed to a feed tank and delivered to the graphite cell and the process continued, as I will proceed now to explain and finally claim.

The accompanying drawing is a diagrammatic elevation illustrating the apparatus in its simplest form, by which the process may be practised.

50 In the drawing, 1 is a feed tank in which the solution to be electrolyzed is placed. This tank is provided with any suitable means 2 for discharging the contents of the tank into the electrolytic cell 3 having an

anode 4 of carbon, such as graphite, and a cathode 5 of iron or other suitable durable material. The anode and cathode in this cell are suitably supplied with an electric current which acting upon the electrolyte therein serves to electrolyze this solution for the partial production of the desired product. The electrolyzed solution overflows from the cell 3 through pipe 6, or other means, to the cell 7, in which the anode 8 is of platinum, or some alloy thereof, and the cathode 9 is of iron or other suitable durable material, and these electrodes in this cell are supplied with electric current in any suitable way, and the electrolysis is continued in such a manner that sufficient heat will be generated by the electric current to accelerate the formation of chlorate to such an extent that the final concentration of the desired product in the electrolyte or solution will be obtained by direct formation and conversion in this cell. For convenience the cell 3 will be herein referred to as the "graphite cell," and the cell 7 as the "platinum cell." From the platinum cell the electrolyte will be removed, as by pipe 10, to a cooling tank 11 in which the product is crystallized. Crystallization may be accelerated by use of any suitable cooling agent, and I have indicated by dotted lines 12, a coil arranged in the bottom of the crystallizing tank which may be supplied with refrigerating brine, or any other suitable cooling agent, circulated therethrough. As the product crystallizes in this tank it is removed, and the mother liquor is taken away, as through pipe 13, to a storage tank 14 and there refreshed or enriched with additional salts. This tank 14 may be connected by a pipe 15 with a pump 16 by which this mother liquor may be returned to the feed tank 1, through the pipe 17, to continue the process.

For the manufacture of potassium chlorate, I use a nearly saturated aqueous solution of potassium chlorid, to which may be added a small quantity of potassium chromate or potassium bichromate for the purpose of improving the efficiency of the subsequent electrolytic conversion. This solution contained in the feed tank is fed continuously therefrom into the graphite cell and the electric current supplied. The temperature of the electrolyte in this cell is

maintained in any suitable way below 35° centigrade, and the electrolysis continued until the electrolyte is nearly saturated, at the temperature of the cell, with potassium chlorate, formed therein by the action of the electric current. The thus electrolyzed solution is then conveyed into the platinum cell and the electric current supplied to that cell, and by action of the current in this platinum cell, the electrolyte is raised to a considerably higher temperature, preferably above 70° centigrade. The effect of the current in the platinum cell is to increase the quantity of chlorate contained in the electrolyte or solution, through its direct action on the potassium chlorid and by efficiently converting to chlorate, the hypochlorite which remained in the liquor coming over from the graphite cell.

It is to be understood, of course, that the invention is not limited to the use of a single graphite cell and a single platinum cell, or to the employment of the same relative numbers of each, but I do esteem it of importance that in all cases the liquor after leaving the final graphite cell shall enter one of the platinum cells. The number of platinum cells will be regulated by the final temperature of the electrolyte which it is desired to obtain. Nor do I limit the invention to the electric connections shown, but it is important that graphite and platinum anode respectively be embodied in the two types of cells. Furthermore, the source of current for each type of cell may be independent.

Among other advantages of my combined process I may mention the following:—

First. A very great saving in the cost of apparatus necessary to manufacture a fixed quantity of chlorate, over the use of platinum anodes alone, since a great part of the necessary electrolysis is conducted with the very much cheaper graphite anodes, thus necessitating a smaller platinum equipment.

Second. The process of subsequent electrolysis with platinum anodes and at a higher temperature proves the most efficient method of converting to chlorates any hypochlorites remaining in the electrolyte which is carried over from those cells which have graphite anodes and which are operated at a lower temperature.

Third. A decided advantage of the combined process over the use of graphite anodes alone is the greater final concentration of chlorate possible in the electrolyte due to the elevated temperature of the subsequent electrolysis in the platinum cell. This results in the handling of much smaller quantities of electrolyte, with its consequent saving of storage

capacities, and the greater ease of cooling the electrolyte for crystallization purposes.

What I claim is:—

1. The process of producing chlorates of alkalis or alkaline earths, which consists in subjecting an aqueous solution of the chlorid of the base whose chlorate is to be formed, to electrolysis in a cell having a graphite anode, at a relatively low temperature, and subsequently further electrolyzing the thus electrolyzed solution in a cell having a platinum anode, at a higher temperature.

2. The process of producing chlorates of alkalis or alkaline earths, which consists in subjecting an aqueous solution of the chlorid of a base whose chlorate is to be formed, to electrolysis in a cell having a graphite anode, at a relatively low temperature, and subsequently further electrolyzing the thus electrolyzed solution in a cell having a platinum anode, at a higher temperature, the higher temperature being produced by the electric current flowing through the cell with the platinum anode.

3. The process of producing chlorates of alkalis or alkaline earths, which consists in subjecting an aqueous solution of the chlorid of a base whose chlorate is to be produced, to a primary electrolysis in a cell having a graphite anode, in the presence of a relatively low temperature, then further electrolyzing the thus electrolyzed solution in a cell having a platinum anode and in which a relatively high heat is maintained, then removing the liquor and crystallizing it.

4. The process of producing potassium chlorate, which consists in primarily electrolyzing an aqueous solution of potassium chlorid in the presence of a graphite anode and at a relatively low temperature, and subsequently further electrolyzing the thus electrolyzed solution in the presence of a platinum anode at a relatively high temperature.

5. The process of producing potassium chlorate, consisting in subjecting an aqueous solution of potassium chlorid and a potassium derivative of chromic acid to electrolysis in the presence of a graphite anode and low temperature, and thereafter further electrolyzing it in the presence of a platinum anode and higher temperature generated in the cell.

In testimony whereof I have hereunto set my hand this tenth day of September A. D. 1914.

WALTER LAIB.

Witnesses:

W. G. BOWMAN,  
A. M. BECK.