No. 859,431. PATENTED JULY 9, 1907.

C. F. CARRIER, Jr.

APPARATUS FOR THE ELECTROLYTIC PRODUCTION OF CHEMICAL COMPOUNDS.

APPLICATION FILED JUNE 9, 1906.

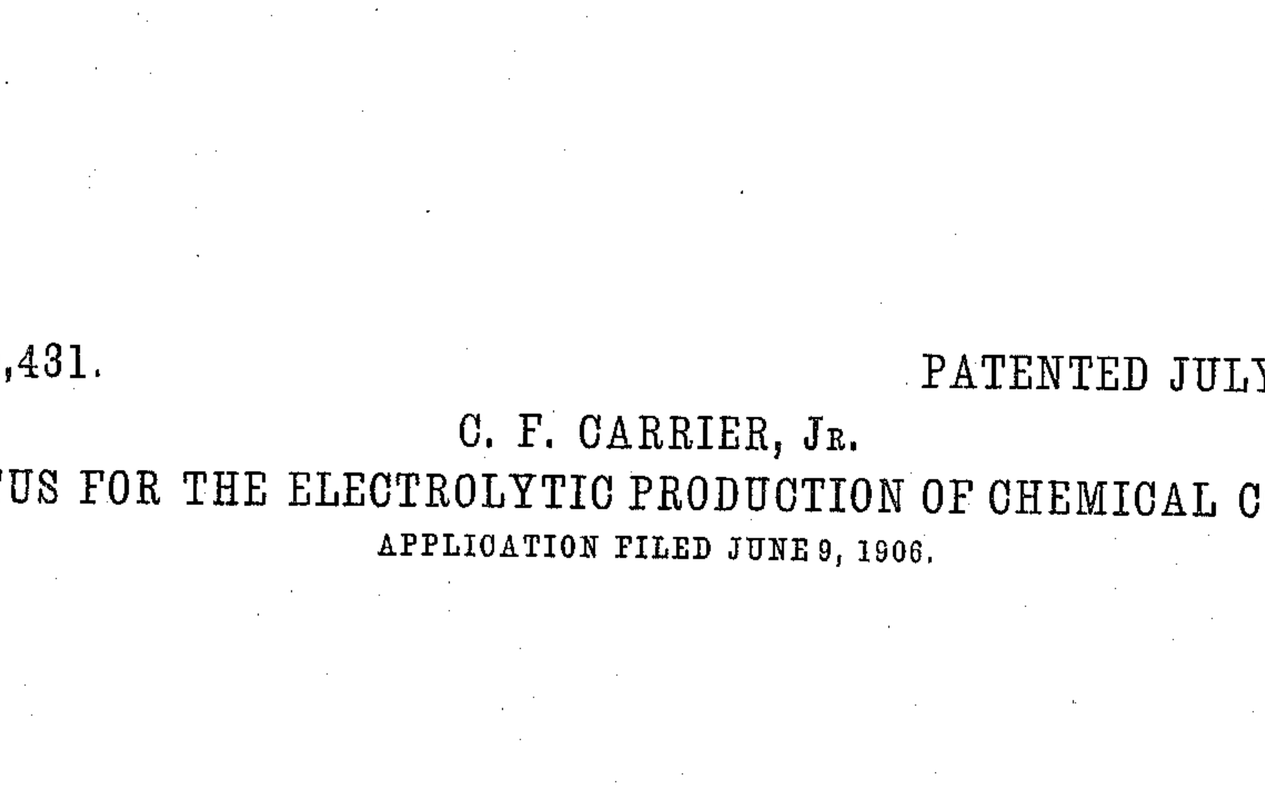

Fig. 1.

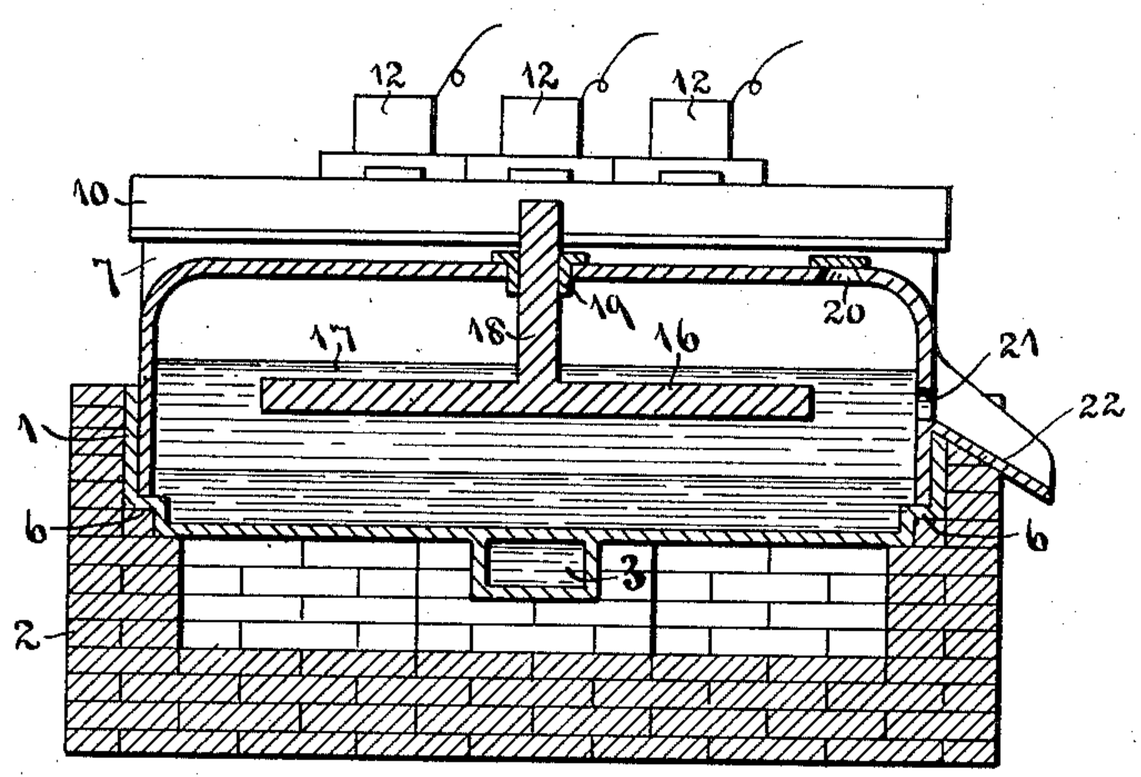

Fig. 2.

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APPARATUS FOR THE ELECTROLYTIC PRODUCTION OF CHEMICAL COMPOUNDS.

No. 859,431. Specification of Letters Patent. Patented July 9, 1907.

Application filed June 9, 1906. Serial No. 320,987.

To all whom it may concern:

Be it known that I, COURTLAND F. CARRIER, JR., a citizen of the United States, residing at Elmira, in the county of Chemung and State of New York, have invented certain new and useful Improvements in Apparatus for the Electrolytic Production of Chemical Compounds, of which the following is a specification.

This invention relates to improvements in apparatus adapted for the production of certain chemical compounds by electrolysis; and applies, more especially, to apparatus for the preparation of those compounds which ordinarily require the use of the free metallic element in their preparation.

The object of my invention is to provide an apparatus of this character, whereby such compounds may be continuously produced, and in a manner much more economical than has hitherto been attained.

The apparatus, and its mode of operation, may be best understood by describing it in connection with the production of the oxids of sodium, although its use is not to be understood as being confined to the production of these specific compounds. As now prepared, the oxids of sodium are produced by the oxidation of previously prepared metallic sodium. By the use of my improved apparatus, however, it is possible to prepare the oxids in a single operation, without using the expensively prepared metallic sodium in its free form. I attain my object by constructing the apparatus substantially in the manner illustrated in the accompanying drawings, in which:—

Figure 1 represents a longitudinal vertical section through the apparatus; and Fig. 2, a transverse vertical section of the same on the line *a*—*a* in Fig. 1.

Like numerals designate like parts in the two views.

A large "pan" 1 set in masonry 2, constitutes the body of the apparatus. This pan is best made of cast iron and is preferably of rectangular form. On the under side of the pan, and cast in one piece with it, is a longitudinal compartment 3, the purpose of which will be hereinafter described. This compartment communicates with the pan through circular openings formed at 4 and 5. The opening 4, in Fig. 1, is shown positioned directly beneath the electrode 12, and where more than one of these electrodes are employed, the chamber 3, at this end, will be so arranged that there will be one of these openings beneath each electrode. The remaining portions of the apparatus are supported within the pan by means of shoulders or lugs formed thereon as indicated at 6.

A frame 7, preferably of cast iron, open at top and bottom, incloses the anode compartment, this frame being lined with basic material 8, such as magnesia, or other material which will not be attacked by the electrolyte 9 contained therein, or by the gases evolved during the process. The compartment is closed at the top by cover 10 composed of material similar to that of the lining. This cover is supplied with feed holes 11 for replenishing the supply of the electrolyte from time to time. Openings are also provided for the anodes 12 which are composed most suitably of graphite. To protect the anodes they are surrounded by sleeves 13 of material capable of withstanding the action of the gases in the compartment. The number of anodes may vary according to the desired capacity of the apparatus. At the back of the anode compartment is a port 14, through which the gases formed during the process are conducted away.

The cathode compartment consists of a rectangular box 15, preferably of cast iron, which is closed upon all sides except the bottom, being in the form of a rectangular bell. The cathode is a flat plate of iron or nickel 16, which is supported in the electrolyte 17 by one or more heavy lugs 18 projecting through the bell and insulated therefrom by sleeves 19 of any suitable insulating material. This cathode compartment is also provided with a charging opening 20 and with a tap hole 21. This tap hole may be closed by a plug and the material drawn therefrom intermittently by the removal of the plug from time to time; or the melted product may be allowed to flow continuously from the hole by way of the channeled lip 22.

At the end of the apparatus beyond the cathode compartment is located a circulating device. This consists of a screw propeller 23, which is rotated in the circular opening 5 leading to the compartment 3 by means of proper power transmission devices, such as the spur gear 24 geared in with a suitable power drive. The propeller shaft 25 may be conveniently supported by means of brackets 26 and 27 secured to, or formed upon, the end of the pan, the propeller being suspended at the proper point in opening 5 by means of a collar 28.

Gas burners 29 are arranged in chambers in the masonry beneath the pan to act as an auxiliary source of heat, and to assist in starting the apparatus, or to tide over an interruption in the electric current.

As an example of the mode of operating this apparatus, the oxids of sodium may be prepared therein as follows: By means of the burners 29, the furnace is heated to over 350° C and lead is melted in the pan, or poured in already melted. Sufficient lead is added to fill the lower compartment 3 and the pan to a depth sufficient to form a liquid seal at the bottom of the electrolytic compartments. Tin, cadmium, zinc, or certain alloys might be used in place of lead, but lead is the cheapest. The cathode compartment is filled to the desired depth with melted sodium hydroxid. A small carbon rod is inserted in one of the graphite anodes in the anode compartment and the anodes are lowered till the rod dips into the lead. When the current is started this rod is heated to bright redness

and, by surrounding it with common salt, it is soon possible to melt enough salt to bring the large graphite anodes into action. When sufficient melted salt has been obtained the carbon rod is broken off and the current passes through the electrolyte, which is thereby decomposed, yielding chlorin at the anodes and metallic sodium at the surface of the lead, which here acts as a cathode.

The chlorin escapes through the port 14 and the sodium is absorbed by the lead, thereby forming an alloy. This alloy is much lighter than pure lead and tends to float on the surface, where there is danger of its being consumed by the electrolyte and any gases in solution. This is prevented by means of a rapid circulation of the lead. The propeller 23 forces the lead down through the opening 5, into and through chamber 3, and thence up through the opening, or openings, 4. The alloy formed in the anode compartment is thus carried on to the cathode compartment as fast as formed, and thus the sodium is prevented from reuniting with the electrolyte 9. There is also a special advantage in causing the circulation as described. Any impurities in the salt will tend to settle to the bottom and coat over the surface of the lead. This increases the resistance and wastes electrical energy, and also decreases the ampere efficiency of the sodium formation. When the lead is forced upward through an opening as shown with sufficient force to form a slight "mound", as at 30, this scum will be forced aside by the currents flowing radially from the mound in all directions, thus leaving a clear lead surface directly below the anode for the reception of the liberated sodium. The same result can be accomplished by carrying the opening 4 above the general level of the lead by means of a pipe, but the force required is greater and the action no more efficient. The inventor is aware that metallic cathodes have been used, where the return current enters from below, but no application has hitherto been made, to his knowledge, of a circulation so strong that the results as above described are accomplished for the purpose set forth.

The sodium-lead alloy is carried beneath the cathode as it circulates in the manner described. In this compartment the lead acts as anode. The passage of the current tends to liberate metallic sodium at the cathode and to dissolve sodium from the sodium-lead alloy at the anode. Under proper conditions metallic sodium may be prepared in this way. If, now, an oxidizing agent is added to the sodium hydroxid bath, no sodium will be liberated, but sodium oxids will be produced as follows:

$$NaNO_3+3NaOH+8Na=6Na_2O+NH_3 \quad (I)$$

$$NaNO_3+3NaOH+2Na=3Na_2O_2+NH_3 \quad (II)$$

$$Na_2O+Air=Na_2O_2+XN$$

Reactions I and II will take place simultaneously, their relative preponderance depending upon the conditions of the operation. The product, as withdrawn from the cathode compartment, will contain both Na_2O and Na_2O_2. The Na_2O may be converted into Na_2O_2 by heating in an oxidizing atmosphere, air free from moisture and carbon dioxid being most suitable. The nitrate is added from time to time in a proportion derived from the above reaction and the number of amperes used. The alloy from which a portion of the sodium has been removed is then returned to the anode compartment in the manner described.

It is to be specially noted that, by the use of this apparatus, it is possible to produce compounds whose production previously involved the use of a free metallic element, without the previous separation of that metal. Also that in this manner it is possible to continuously produce a larger quantity of end product than could be produced if the same number of amperes were to be employed in the previous production of the metal. For example, to produce six molecules of Na_2O by the old method requires 12 atoms of metallic Na. This new apparatus makes it possible to produce the same quantity of Na_2O with a consumption of current equivalent to that required to produce eight atoms of Na.

Having thus described my apparatus and its mode of operation, but without confining it to the production of any one of the particular class of compounds hereinabove described, nor to the production of such compounds alone, since, by properly arranging the cathode compartment, a metal in its free state may also be produced and drawn therefrom; what I claim as new and desire to secure by Letters-Patent is:

1. An electrolytic apparatus comprising anode and cathode compartments, a bath of molten metal common to both compartments, circulating passages whereby the metal of the bath will be conducted from the anode compartment to the cathode compartment and return, the return passage being provided with an orifice leading into the anode compartment directly beneath an electrode therein, and means for producing a circulation of the metal through said passages whereby an upheaval of the bath will be caused in the anode compartment directly beneath the electrode.

2. An electrolytic apparatus comprising anode and cathode compartments having their bottom ends open and sealed by a bath of molten metal common to both, a return channel leading from the side of the cathode compartment remote from the anode compartment to a point in the anode compartment directly beneath an electrode therein and means for producing a circulation of the metal of the bath through said channel whereby an upheaval of the bath will be caused directly beneath said electrode.

3. An electrolytic apparatus comprising a pan adapted to contain a molten metal bath, anode and cathode compartments having their bottom ends open and supported above the bottom of the pan in position to be sealed by the metal of the bath, a channel formed on the under side of the pan leading from a point beyond the cathode compartment to a point below an electrode in the anode compartment, openings in the bottom of the pan leading to said channel at said points, a screw propeller mounted to rotate in the opening beyond the cathode compartment, means for rotating the propeller, means for charging the compartments, and means for drawing off the end product from the cathode compartment.

In testimony whereof I have affixed my signature, in presence of two witnesses.

COURTLAND F. CARRIER, JR.

Witnesses:
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