No. 809,089. PATENTED JAN. 2, 1906.

H. S. BLACKMORE.

PROCESS OF MAKING CAUSTIC ALKALI.

APPLICATION FILED MAY 10, 1905.

Fig. 1

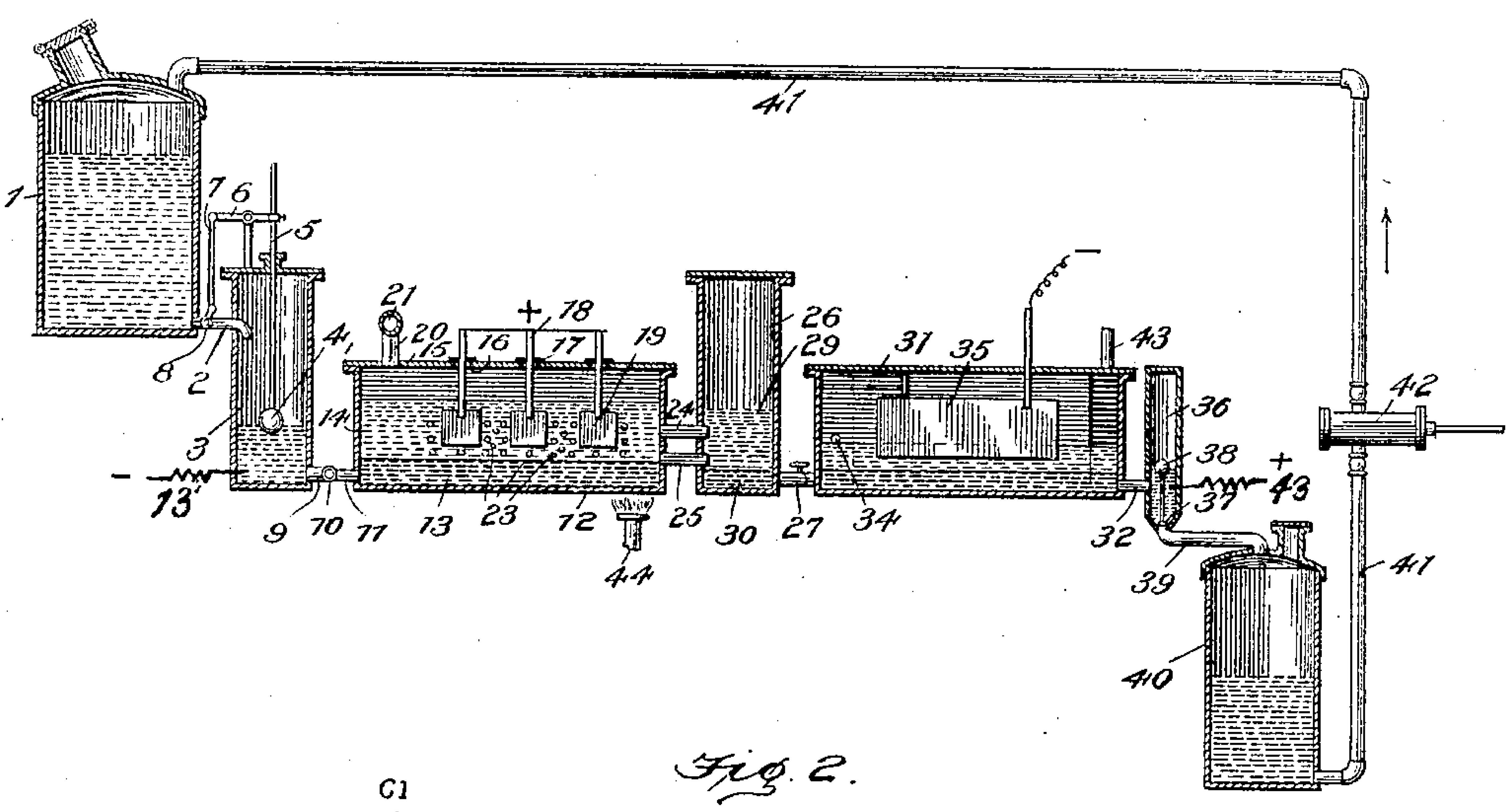


Fig. 2.

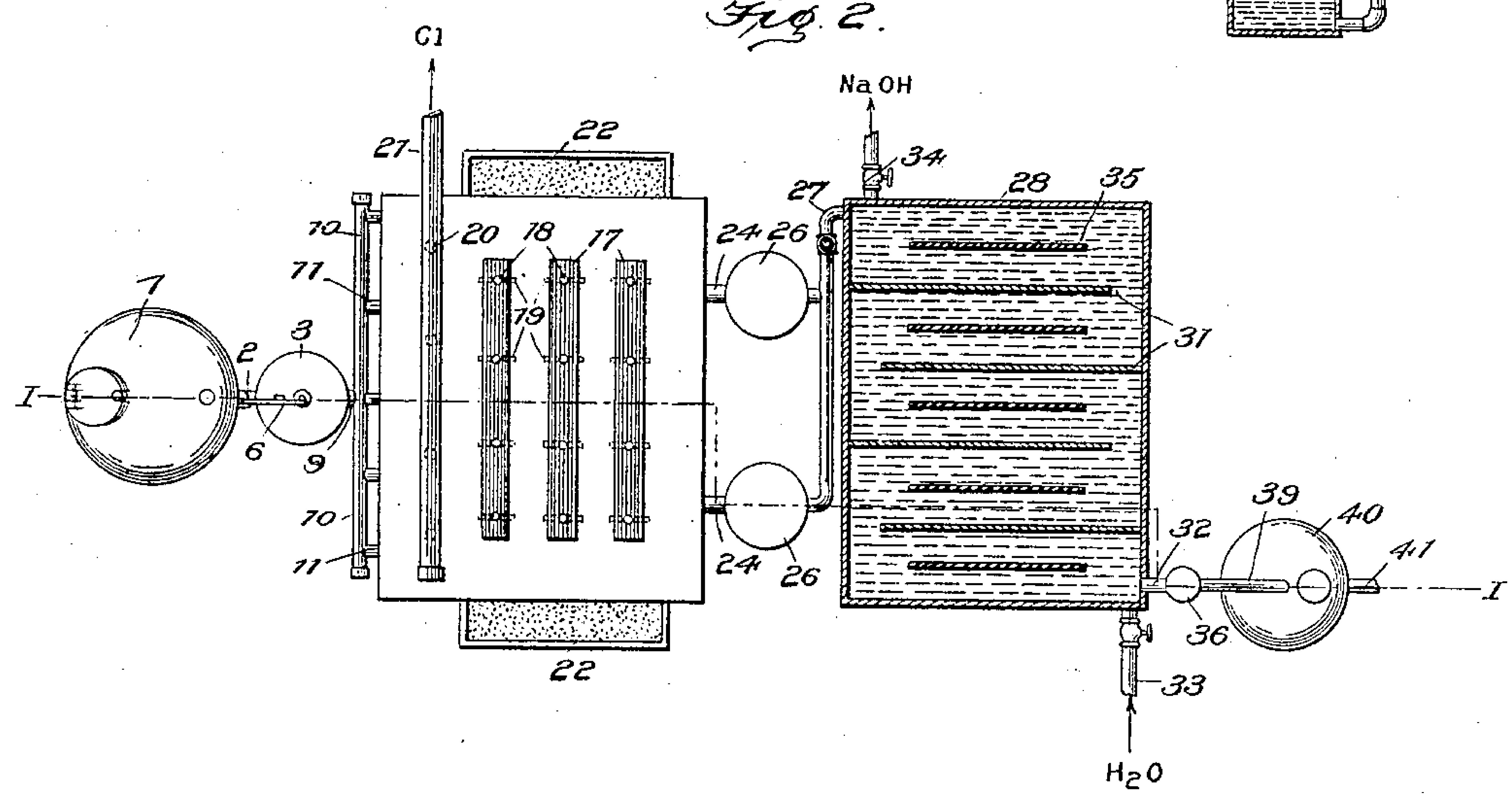
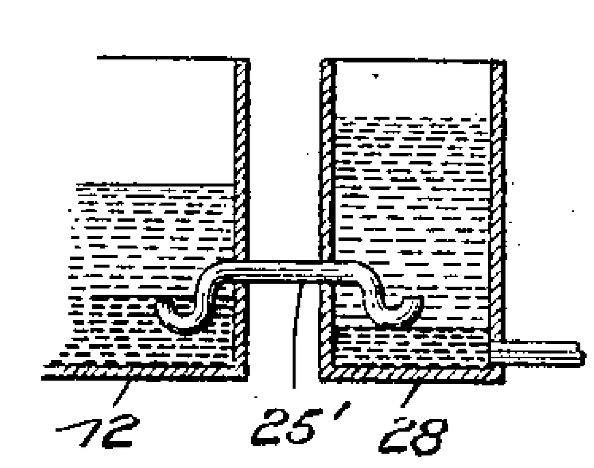


Fig. 3.



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PROCESS OF MAKING CAUSTIC ALKALI.

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Application filed May 10, 1905. Serial No. 259,819.

To all whom it may concern:

Be it known that I, HENRY SPENCER BLACKMORE, a citizen of the United States, residing at Mount Vernon, in the county of Westchester and State of New York, have invented certain new and useful Improvements in Processes of Making Caustic Alkali, of which the following is a specification.

The object of my invention is to make caustic alkali by electrolytic action in such a manner as to effect a saving in time and expense together with a larger yield with less power in an automatic manner than has been obtained hitherto; and it consists of certain new and novel features, as hereinafter set forth.

My invention relates particularly to the production of caustic soda and chlorin from aqueous solutions of common salt, but is not limited thereto, as it may be applied to the production of many other alkalis, such as potassa or lithia and other salts or compounds of alkali than the chlorid, such as sodium chlorid, as employed in the specific example of my process herein set forth.

In carrying out my invention for the production of caustic soda from an aqueous solution of common salt (sodium chlorid) I proceed as follows, reference being had to the accompanying drawings, in which—

Figure 1 is a longitudinal vertical section of the apparatus, taken on line I I of Fig. 2. Fig. 2 is a plan view, partly in horizontal section; and Fig. 3 illustrates a modification, using a siphon 25′ to draw off the alloy from the electrolytic cell, a body of liquid other than the electrolyte being used in the intermediate vessel 28—for example, carbon bisulfid, carbon tetrachlorid, &c.

The apparatus consists of the following parts: a vessel 1 communicates by bottom outlet 2 with a chamber 3. Within chamber 3 is a ball-float 4, communicating by rod 5, lever 6, and link 7 with a valve 8 in outlet 2. This float-feed mechanism serves to maintain a column of mercury at uniform level in chamber 3. An outlet-pipe 9, leading from the bottom of chamber 3, opens into a manifold 10, which in turn has several outlet-pipes 11, opening into the bottom of electrolytic cell 12. The layer of mercury 13 thus introduced upon the bottom of electrolytic cell is connected to act as cathode by contact 13′. Supported upon cathode 13 is a body of an aqueous solution of sodium chlorid or other electrolyte 14. The cover 15 of the electrolytic cell has a number of openings 16, arranged in parallel rows. Above each row of openings is a strip 17 of insulating material which serves to support the stems 18 of anodes 19. The cover 15 has also a number of vertical outlet-pipes 20 for chlorin leading into a manifold. Upon each end of the electrolytic cell is arranged a receptacle 22 for solid sodium chlorid or other substances to be electrolyzed. These receptacles are in open communication with the electrolyte in cell 13 by openings 23 and serve to maintain it in a saturated condition. From the side of cell 13 one or more sets of pipes 24 25 lead into one or more vessels 26, from the bottom of which in turn a valve-pipe 27 leads into the oxidizing vessel 28. Pipe 24 serves to put the electrolyte into open communication with a similar body of liquid 29 in vessel 26, while pipe 25 is arranged to draw off the liquid amalgam produced by electrolysis as soon as the same rises above the normal level of the cathode.

The oxidizing vessel 28 is divided into a number of parallel compartments alternately communicating at opposite ends by vertical baffle-plates 31. The liquid amalgam is introduced by pipe 27 into one end of the compartments and flows back and forth through the succeeding compartments to an inlet-pipe 32. Adjacent to but above outlet 32 is an inlet-pipe 33 for water or other oxidizing agents. The introduced water flows over the surface of the amalgam and serves to remove therefrom the sodium or other easily-oxidizable metals, the resulting sodium hydrate escaping through the valved outlet-pipe 34. The rate of oxidation of the sodium may be increased by placing electrodes 35, of carbon or other relatively electronegative material, in the water and putting them in electrical communication with the amalgam below either directly or by connecting the amalgam with the positive lead of the electrolyzing-current 43 and the carbon electrodes with the negative lead, thereby utilizing the electrical energy generated in the oxidizing-cell to furnish current to the electrolyzing-cell. Pipe 32 serves to carry the depleted alloy metal from the oxidizing-cell into an adjoining vertical cylinder 36, in the bottom of which is a valve-outlet 37, controlled by a float 38. A pipe 39 leads from outlet 37 into a receiving vessel 40. The mercury collected in vessel 40 is returned by pipes 41, including pump 42, or otherwise to the reservoir 1. The movement of mercury and amalgam through the various vessels is effected solely by the differences of level of the various bodies of mercury and amalgam, and these differences are maintained by suitable predetermined adjustment of float-feed 8 and float-controlled outlet 37 and of the height of the connecting-pipes. The connecting-pipes between the various vessels are preferably of hard rubber or vitrified earthenware to insulate the vessels from each other. The pipe 21 for removing chlorin is preferably connected with an exhaust device to maintain a slight vacuum over the electrolyte and facilitate the escape of chlorin. The maintenance of the electrolyte in a saturated condition tends to prevent the absorption of chlorin by it and prevents the changes of level which would otherwise occur. The outlet-pipe 34 for sodium hydrate should be somewhat larger than the water-inlet pipe 33 and serves to maintain a constant level of the water in the oxidizing-chambers.

By the use of vessels which are provided with heating means the apparatus may be adapted for the electrolysis of molten sodium chlorid upon a cathode of molten metal, such as lead, any suitable means being employed for oxidizing and removing the sodium, such as a stream of molten sodium hydrate flowing in contact with the lead-sodium alloy or injected into the body of the alloy, the resulting sodium oxid being regenerated, if desired, by the action of steam.

The term "alloy," as used in the claims is intended to cover a solution of an oxidizable metal in any alloying metal, whether mercury or lead.

As is well known, the addition of a comparatively small amount of alkali or an alkaline-earth metal to mercury converts the latter into a pasty condition in which it does not readily flow. This diffiulty may be avoided when mercury is employed as a cathode by applying heat to it, thereby maintaining the amalgam produced by electrolysis in fluent condition and enabling larger percentages of sodium to be absorbed without interforcing with the continuous overflow of amalgam into the oxidizing vessel. Such heat may be supplied by a gas-burner 44 beneath the electrolytic cell, as indicated by dotted lines in Fig. 1.

It will be noted that the alkali-metal amalgam or alloy produced in the electrolytic vessel or cell 12 is at a higher level than the liquid metal in the oxidizing vessel or cell 28 and that metallic connection between the metal in the electrolytic vessel and the oxidizing vessel is prevented by the interposition of a liquid compound which is either a non-conductor of electricity, such as carbon bisulfid or carbon tetrachlorid, or a liquid or a liquefied compound not a conductor of the first degree to the electric current employed in the electrolytic vessel for the purpose of electrolysis, by which means the electric current is prevented from short-circuiting between the liquid metal in the oxidizing vessel, which is electrically connected as cathode, and the liquid metal in the oxidizing-chamber, which is electrically connected as anode. It will also be noted that the sodium amalgam passing from the electrolytic vessel as it is formed into the oxidizing vessel through a liquid breaking its metallic connection admits of the alkali-metal amalgam passing from one vessel to the other in such a manner that the short-circuiting of electric currents is prevented, and also being accomplished below the surface of the metal contact breaking liquid compound the oxidation of the mercury which would otherwise occur in the presence of atmospheric oxygen is prevented.

In carrying out my invention for producing caustic soda from common salt (sodium chlorid) I place in the electrolytic vessel 12 a quantity of water and saturate the same with common salt, (sodium chlorid,) which is dissolved into the water from the receptacles 22, in which it is placed. I also place water in vessel 28 and introduce below the same a layer or stratum of mercury. I then place in vessel 26 upon the mercury therein a quantity of carbon tetrachlorid sufficient to reach the connecting-pipe 25 and then feed mercury from receptacle or reservoir 1 through the metal-regulating chamber or balancing-chamber 3 into the electrolytic cell 12 until it nearly reaches the outlet-pipe 25, the float check-valve in vessel 3 being regulated to check the flow of mercury at that level. I then pass a current of electricity through the anodes 19, connecting the mercury in the bottom of electrolytic vessel 12 as cathode, whereupon the sodium chlorid in solution above the mercury cathode becomes electrolyzed and chlorin and sodium liberated therefrom. The chlorin liberated at the anodes passes up and out through the outlet-pipes 20 and 21, while the sodium unites superficially with the mercury, and thus increases the bulk thereof until it rises above the level of the outlet-pipe 25, whereupon it overflows and drops through the layer of carbon tetrachlorid in vessel 26 and accumulates and passes out into the oxidizing vessel 28 through the connecting-pipe 27. In the oxidizing vessel this sodium amalgam is connected as anode, while suspended in the weak solution of caustic soda floating upon the sodium amalgam is the copper cathode 35. A current of electricity is then passed through the cathode 35 and the liquid-metal anode, whereupon the sodium therein becomes oxidized and is separated therefrom, dissolving in the weak alkali solution, while the mercury depleted of its sodium as it accumulates gradually raises the valve 38 and passes out into the cistern or reservoir 40, from which it is returned to reservoir 1 to be reused.

During the continuous operation of the process it will be seen that the whole process is operated in an automatic manner by gravity actuated by the increase and decrease in bulk of the liquid metal actuated by electrolytic action.

The caustic soda produced in the oxidizing-chamber 28 is gradually withdrawn through the pipe 34, controlled by a valve therein, while a corresponding amount of water is allowed to pass in and over the mercury amalgam during electrolytic action through pipe 33. In this manner the water is gradually passed through the oxidizing-cell over the sodium amalgam, the sodium in which is being oxidized and dissolved in water with the formation of caustic soda in a tortuous manner and with such a speed with relation to electrolytic action in the cell that a solution of caustic soda of calculated strength may be withdrawn through pipe 34 accordingly as the water is supplied through pipe 33.

Having now described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of making caustic alkali from salts of alkali-metals, which consists in introducing into a cell containing an aqueous solution of the alkali metal salt to be decomposed, a liquid metal, liberating the alkali metal in juxtaposition to the liquid metal by electrolysis, causing the liquid metal containing the alkali metal to pass into a separate or oxidizing cell through a liquid compound breaking its metallic connection between the liquid metal in the electrolytic cell and the liquid metal in the oxidizing-cell, by gravity, oxidizing the alkali metal contained in the liquid metal in the oxidizing-cell with the formation of caustic alkali, by electrolysis, and causing the liquid metal depleted of alkali to pass from the oxidizing-cell by gravity.

2. The process of making caustic alkali from salts of alkali metals, which consists in passing a liquid metal through an electrolytic cell containing an aqueous solution of the alkali-metal salt to be decomposed during electrolysis, decreasing the density and increasing the bulk of the liquid metal in the said electrolytic cell by causing it to unite with the alkali metal liberated by electrolysis therein, and causing it to overflow into a separate or oxidizing cell through a liquid compound capable of breaking metallic contact of the liquid metal in the separate cells from each other, by gravity, increasing the density of the liquid metal in the oxidizing-cell by separating the alkali metal therefrom by electrolytic oxidation, and discharging the liquid metal, thus separated from alkali metal, therefrom, by gravity.

3. The process of making caustic soda from sodium compounds, which consists in introducing into a cell containing an aqueous solution of the sodium compound to be decomposed, a liquid metal, liberating the sodium in juxtaposition to the liquid metal by electrolysis, causing the liquid metal containing the sodium to pass into a separate or oxidizing cell through a liquid compound breaking its metallic connection between the liquid metal in the electrolytic cell, and the liquid metal in the oxidizing-cell, by gravity, oxidizing the sodium contained in the liquid metal in the oxidizing-cell with the formation of caustic soda by electrolysis and causing the liquid metal depleted of sodium to pass from the oxidizing-cell, by gravity.

4. The process of making caustic soda from sodium chlorid, which consists in introducing into a cell containing an aqueous solution of the sodium chlorid to be decomposed, a liquid metal, liberating the sodium in juxtaposition to the liquid metal by electrolysis, causing the liquid metal containing the sodium to pass into a separate or oxidizing cell through a liquid compound breaking its metallic connection between the liquid metal in the electrolytic cell, and the liquid metal in the oxidizing-cell, by gravity, oxidizing the sodium contained in the liquid metal in the oxidizing-cell with the formation of caustic soda by electrolysis and causing the liquid metal depleted of sodium to pass from the oxidizing-cell, by gravity.

5. The process of making caustic alkali from salts of alkali metal, which consists in introducing mercury into a cell containing an aqueous solution of the alkali-metal salt to be decomposed, liberating the alkali metal in juxtaposition to the mercury, causing the mercury containing the alkali metal to pass into a separate or oxidizing cell through a liquid compound breaking its metallic connection between the mercury in the electrolytic cell and the mercury in the oxidizing-cell, by gravity, oxidizing the alkali metal contained in the mercury in the oxidizing-cell with the formation of caustic alkali, by electrolysis, and causing the mercury depleted of alkali to pass from the oxidizing-cell by gravity.

6. The process of making caustic soda from sodium compounds, which consists in introducing mercury into a cell containing an aqueous solution of the sodium compound to be decomposed, liberating the sodium in juxtaposition to the mercury by electrolysis, causing the mercury containing the sodium to pass into a separate or oxidizing cell through a liquid compound breaking its metallic connection between the mercury in the electrolytic cell, and the mercury in the oxidizing-cell, by gravity, oxidizing the sodium contained in the mercury in the oxidizing-cell with the formation of caustic soda by electrolysis and causing the mercury depleted of sodium to pass from the oxidizing-cell, by gravity.

7. The process of making caustic soda from sodium chlorid, which consists in introducing mercury into a cell containing an aqueous solution of the sodium chlorid to be decomposed, liberating the sodium in juxtaposition to the mercury by electrolysis, causing the mercury containing the sodium to pass into a separate or oxidizing cell through a liquid compound

breaking its metallic connection between the mercury in the electrolytic cell, and the mercury in the oxidizing-cell, by gravity, oxidizing the sodium contained in the mercury in the oxidizing-cell with the formation of caustic soda by electrolysis and causing the mercury depleted of sodium to pass from the oxidizing-cell, by gravity.

8. The process of making caustic alkali from salts of alkali metals, which consists in passing mercury through an electrolytic cell containing an aqueous solution of the alkali-metal salt to be decomposed during electrolysis, decreasing the density and increasing the bulk of the mercury in the said electrolytic cell by causing it to unite with the alkali metal liberated by electrolysis therein, and causing it to overflow into a separate or oxidizing cell through a liquid compound capable of breaking metallic contact of the mercury in the separate cells from each other, by gravity, increasing the density of the mercury in the oxidizing-cell by separating the alkali metal therefrom by electrolytic oxidation, and discharging the mercury, thus separated from alkali metal, by gravity.

9. The process of making caustic alkali from salts of alkali metals, which consists in passing mercury through an electrolytic cell containing an aqueous solution of an alkali-metal salt to be decomposed during electrolysis, decreasing the density and increasing the bulk of the mercury in the said electrolytic cell by causing it to unite with the alkali metal liberated by electrolysis therein, causing it to overflow into a separate or oxidizing cell through an electrically non-conductive liquid capable of breaking metallic contact of the mercury in the separate cells from each other, by gravity, increasing the density of the mercury in the oxidizing-cell by separating the alkali metal therefrom by electrolytic oxidation, and discharging the mercury, thus separated from the alkali metal, therefrom, by gravity.

10. The process of making caustic alkali from salts of alkali metals, which consists in passing mercury through an electrolytic cell containing an aqueous solution of an alkali-metal salt to be decomposed during electrolysis, decreasing the density and increasing the bulk of the mercury in the said electrolytic cell by causing it to unite with the alkali metal liberated by electrolysis therein, causing it to overflow into a separate or oxidizing cell through carbon tetrachlorid thereby breaking metallic contact of the mercury in the separate cells from each other, increasing the density of the mercury in the oxidizing-cell by separating the alkali metal therefrom by electrolytic oxidation, and discharging the mercury, thus separated from the alkali metal, by gravity.

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

HENRY SPENCER BLACKMORE.

Witnesses:
H. N. JENKINS,
ALBERT W. SIOUSSA.