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PROCESS OF RECOVERING ALKALI METALS AND BY-PRODUCTS

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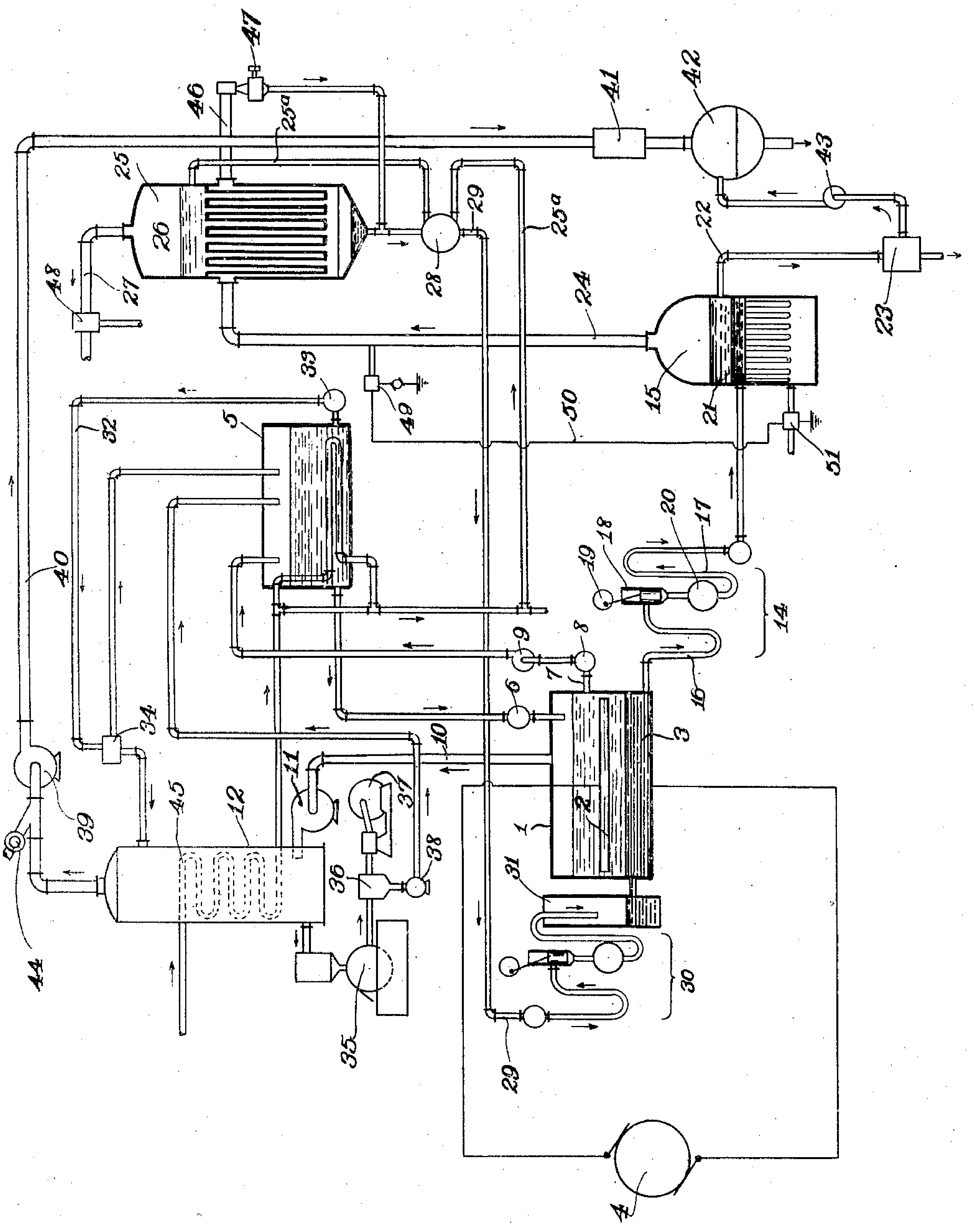

INVENTOR.

Seth A. Moulton

BY

Cornelius Zabriskie

ATTORNEY.

UNITED STATES PATENT OFFICE

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PROCESS OF RECOVERING ALKALI METALS AND BY-PRODUCTS

Seth A. Moulton, Los Angeles, Calif., assignor to Peter F. Crahan, New York, N. Y.

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This invention relates to a novel and useful process for the separation and recovery of alkali metals whose salts are soluble and form an electrolyte and which metals are of such character that they are susceptible to, or can be made to form an amalgam with mercury. For example, the invention is useful primarily for the recovery of metallic sodium and by-products or extended products resulting from the products of the process, but this invention is not confined to sodium as it is applicable to all of the group of alkali metals and metals of the alkali earths, which will amalgamate with mercury and which have melting points below and boiling points above the decomposition points of the respective amalgams.

Commercial metallic sodium is now produced almost exclusively by the electrolysis of molten sodium chloride and molten sodium hydroxide. The processes employed are expensive and destructive to apparatus due to the high temperatures involved. The melting temperature of sodium hydroxide is about 604° F. while the melting point of sodium chloride is about 1481° F.

The most recent accomplishment and that which is considered commercially the best process prior to this invention provides for the electrolysis of fused anhydrous sodium chloride, said process being considered an advance over prior practice on account of the difference in cost between sodium hydroxide and sodium chloride. The benefit of the cheaper raw material, i. e., sodium chloride, however, is materially offset by the cost of refining and purifying. In both the molten sodium hydroxide and molten sodium chloride electrolytic cells, difficulties are encountered in separating the anodic and cathodic products. In the molten sodium chloride cell, the proximity of the melting point of sodium chloride (1481° F.) and the boiling point of metallic sodium (1616° F.) demands very accurate temperature control which, in practice, is difficult to maintain. Also at these temperatures metallic sodium exerts an appreciable vapor pressure which tends to cause considerable metal loss by vaporization. There are other commercial difficulties which are well recognized in the art, but for which, prior to this invention, there have been no satisfactory solutions.

It has also been heretofore proposed to decompose various materials by electrolysis through the employment of electrolytic cells. These electrolytic processes have generally been carried on in connection with the use of electrolysis involving the chlorides, which electrolytic action has resulted in the liberation of chlorine gas, which produces marked deterioration in the apparatus used. Sodium chloride and potassium chloride have been widely used in this connection. The disadvantages resulting are the contamination of the mercury cathode, the necessity of collecting and utilizing the chlorine gas generated and which cannot be discharged into the atmosphere with impunity, and the consumption of the graphite anodes which are generally employed in this connection. The wearing away of the graphite anodes results in variations in the distance between the poles of the cell causing an increase in cell voltage and a decrease in the energy efficiency and also results in the breaking off of graphite particles which collect upon the mercury cathode, causing the evolution of hydrogen gas which is highly detrimental to efficient cell operation.

Furthermore, in such processes as specified, the amalgam-mercury cathode cannot be completely rejuvenated in denuding chambers before it is returned to the decomposition chamber, because too highly purified mercury is susceptible to attack by the dissolved chlorine in the electrolyte, forming mercury chloride before the mercury has become sufficiently charged with sodium.

With the foregoing considerations in mind, the primary object of the present invention is to provide a process which is free or substantially free from the objections which have been stated and at the same time to produce a process which will be highly economical and more efficient in its operation than the prior art.

More specifically, one object of this invention is to produce substantially pure alkali metal by a continuous process, so that one of the end products of the process is an alkali metal in pure or in substantially pure state.

Another object of the invention is to obtain valuable by-products, and if it is desired, to carry the method a step forward and convert the pure metallic alkali metal into a further product herein more specifically set forth.

Another object of the invention is to provide an apparatus whereby the electrolysis of the material operated upon may be effected at relatively low workable temperatures in an efficient manner.

More specifically one object of the invention consists in the treatment of a carbonate solution of an alkali metal, more specifically sodium carbonate, to produce the alkali metal or oxides, more specifically metallic sodium and/or sodium peroxide and sodium bicarbonate.

A further object of the invention relates to the production of a novel apparatus for carrying out the process as a whole, as well as specific steps of the process.

The basic principle of the present process for the recovery of alkali metals and alkali earth metals is founded on the fact that there is a wide divergence between the boiling points of the pure alkali metals and the decomposition points of the respective amalgams; and that there is a corresponding divergence between the melting points of the pure alkali metals and the decomposition points of the respective amalgams, at which latter point mercury is vaporized leaving the pure alkali metal in a molten state.

Speaking generally, the invention consists primarily in a process of electrolyzing a solution of a soluble carbonate of an alkali metal in the presence of mercury, used as a cathode, to form an amalgam. The resulting dilute amalgam-mercury mixture is thereafter heated to a temperature sufficient to volatilize the entire mercury content, but insufficient to volatilize the alkali metal content whereby the amalgam is decomposed to free the alkali metal therefrom and cause the same to form a stratum. During the formation of this stratum, the mercury content of the amalgam goes off in the form of vapor or gas. The alkali metal is decanted or otherwise removed from the zone of heat in pure or substantially pure state. The mercury vapors are condensed for re-use in the cell.

The gases generated in the cell are led therefrom and are useful for the formation of by-products, while, if desired, the alkali metal which constitutes the end product may be subjected to a further treatment in whole or in part to produce a further end product, such, for example, as a peroxide.

From the standpoint of the apparatus employed, the invention, in its preferred form, embodies an electrolytic cell of novel characteristics, energized by an appropriate electrical current and provided with a mercury cathode. The electrolyte, mercury and amalgam are fed into and removed from the cell intermittently to preclude short circuiting. The apparatus further consists in means for feeding the amalgam to a suitable place of heat treatment to bring about the volatilization of the mercury content of the amalgam and to permit the extraction of the alkali metal product.

Further provision is made to recover the mercury by condensation or otherwise, so that the resulting mercury may be fed back to the cell for re-use, while the heat evolved during its condensation is utilized for the generation of steam. Means are further incorporated in the system for receiving the gases evolved in the cell and treating the same by absorption or otherwise to produce such by products as may be desired.

The apparatus of this invention embodies numerous novel features which will be hereinafter more fully described and claimed.

Features of the invention, other than those specified, will be apparent from the hereinafter detailed description and claims, when read in conjunction with the accompanying drawing.

The accompanying drawing illustrates diagrammatically one form of novel apparatus for carrying out the process of this invention which apparatus forms a part of the invention. I wish it understood, however, that the process may be carried out by other specific forms of apparatus than that shown.

In the apparatus shown, no attempt has been made to illustrate thereon the details of any equipment or the relative proportions of the apparatus, as this will be understood by those skilled in the art.

While the practice of the process of this invention may be carried on in connection with various alkali metals, I have chosen to specifically describe the same as applied to the decomposition of sodium carbonate to produce as end products, pure metallic sodium, and/or metallic sodium and sodium peroxide, and sodium bicarbonate.

The specific description will therefore deal with the invention in this connection, it being understood that similar alkali salts and/or compounds may be employed in a like manner.

Referring to the drawing, 1 designates an electrolytic cell provided therein with a metal anode 2 made of any suitable metal which may be plated, for example, with nickel or platnium. 3 designates a mercury cathode formed by a pool of mercury in the cell. Direct current for energizing the cell may be conveniently fed from a direct current generator 4 through appropriate wiring connections. A sodium carbonate solution is adapted to be contained in a tank 5, the brine in this tank being heated in any suitable manner, such, for example, as by internal steam coils to form a substantially saturated solution of sodium carbonate decahydrate. Brine from tank 5 is adapted to be fed through an interrupter 6 into the electrolytic cell 1. This interrupter 6 is of such character as to feed the brine intermittently, but in a substantially continuous way without short circuiting the cell. Any standard form of apparatus for so feeding the brine may be employed. Of course a plurality of cells in series may be used and the brine may be fed from tank 5 to a battery of them, but, for the purpose of illustration, a single cell is shown. The feed of brine to the cell is substantially continuous and the electrolyte is kept at a practically constant level by the overflow pipe 7 through which the depleted brine leaves the cell and passes through an interrupter 8 and a pump 9 back to the tank 5. The brine and tank 5 is kept to a standard strength by appropriate additions of sodium carbonate or sodium carbonate monohydrate.

In operating, the stratum of brine between the poles 2 and 3 of the cell is partially decomposed, causing the evolved carbon dioxide and oxygen gass to bubble up through the electrolyte and be withdrawn from the cell through a pipe 10 including a suction blower 11 which feeds these gases into a scrubber 12, wherein the gases are further treated as hereinafter more fully explained.

The metallic sodium liberated between the poles combines with the mercury cathode 3 to form a fluid dilute mercury-amalgam mixture. This mercury-amalgam mixture is adapted to be fed through an interrupter, designated generally by the reference character 14, to a denuder 15 which may conveniently be in the form of a still. The purpose of the interrupter 14 is to pass the amalgam mixture out of the cell without short circuiting the cell, i. e., to remove the amalgam mixture in such a way that there is not a continuous electrical circuit established between the cell and the still. This interrupter embodies a pair of U-tubes 16 and 17 between which is interposed a pulsating valve 18 in the form of a piston operated by a pitman connected to a crank disk 19 which when rotated causes the piston to reciprocate.

In one leg of the U-tube 17 is an enlarged chamber 20 containing oil or other insulating fluid, the volume of the chamber being greater than the discharge volume of the pulsating valve 18. As a result of this construction, the elevation of the piston will permit amalgam mixture to flow from the cell through the U-tube 16 and through the valve 18 into the chamber 20. When the piston descends, it shuts off the flow of amalgam mixture from the U-tube 16 and the oil in the chamber, which because of difference in specific gravities has remained on the top of the amalgam-mixture in the chamber, serves upon the further downward movement of the piston as a liquid piston to force the amalgam mixture out of the U-tube 17 and permit the same to flow by gravity to the still 15. It will thus be noted that by this apparatus the amalgam mixture is fed intermittently so as not to give a continuous column of amalgam mixture which would serve to short circuit the cell.

The denuder still 15 is heated by any convenient method to such a temperature that it will act upon the amalgam therein to disintegrate the same and yet sufficiently low as not to volatilize the sodium content. As a result, the amalgam is broken up into liquid sodium and mercury vapor. The liquid sodium forms in a stratum designated 21 upon the surface of the amalgam mercury mixture and may be decanted off through a tail pipe 22 to a suitable storage reservoir 23 which may be maintained at any appropriate temperature to keep the metallic sodium in liquid form for packing or for further treatment in the plant.

To operate at the lowest practical temperatures and also to preclude escape of mercury vapors, the denuder still or evaporator is preferably operated under a vacuum and when thus operated, the decanted metallic sodium, collected in the hot well or reservoir 23, acts as a seal for the barometric leg of the decanting pipe. It will of course be understood that the temperatures applied in the still or evaporator will vary in accordance with the degree of vacuum therein, but the material operated upon is such that there are no critical temperatures involved. For example, at atmospheric pressure, metallic sodium has a melting point of about 207° F. and a boiling point of about 1616° F. Mercury at the same pressure and temperature has a melting point of minus 5° F. and a boiling point of 675° F.

When a mixture of mercury and amalgam is heated at a given pressure to a point sufficient to voltatilize the entire mercury content and leave the alkali metal, the final temperature at which the amalgamated mercury is volatilized is dependent mainly on the decomposition temperature of the amalgam, which is generally higher than the boiling point of pure mercury at the given pressure. This decomposition temperature is, however, usually only slightly higher than the boiling point of the pure mercury at the same pressure and relatively far removed from the boiling point of the pure metal so that on the decomposition of the amalgam, there will be formed a pool of molten sodium on the surface of the amalgam or amalgam mixture through which the mercury vapors will escape and this step of the process may be carried out without critical temperatures of heat, because the wide divergence in melting and boiling points of the respective components involved tolerates working pressures and temperatures far removed from the critical.

The mercury volatilized and in the still bubbles up through the sodium stratum 21 and passes through a pipe 24 to a condenser 25 of any conventional form. This condenser, however, is shown as one of the tube or boiler type adapted to contain water within the tubes. The mercury vapors are circulated about the tubes and lose sufficient heat content therein to form steam in the pressure head 26 of the condenser. This steam may be fed through an outlet pipe 27 to do useful work. The mercury condensed in the condenser 25 flows by gravity into a heat exchanger and washer 28 which serves as a source of supply for the cell cathode which also serves to preheat feed water for the condenser 25 fed through pipes 25*a*. The rejuvenated mercury is fed from washer 28 through a pipe 29 to an interrupter 30 which operates in substantially the same manner as the interrupter 14 and serves to feed the rejuvenated mercury into a supply tank 31 from whence it is fed by gravity to the cell. The feed of mercury to the cell and the discharge of amalgam mixture out of the cell is accomplished in a synchronized manner, so as to maintain a substantially uniform pool of mercury in the cell to function as the cathode thereof.

It will be understood that inasmuch as the mercury is fed from the condenser or heat exchanger 25, the mercury enters the washer 28 and is fed back into the cell in a heated condition to accelerate the electrolytic action. By way of example, and without limiting the invention, I may state that in practice the mercury entering the cell may conventiently be in the neighborhood of 200° F. which is a satisfactory operating temperature to obtain a constant state of efficiency. This temperature of 200° F. is above the transition point of the hydrate forming salt in the electrolyte, and, accordingly, salt accumulations do not form on the cell parts. If the temperature were below the transition point, these salt accumulations would occur and destroy the commercial utility of the cell.

As hereinbefore stated, the gases evolved by the electrolytic action in the cell and in the present instance, carbon dioxide and oxygen gas pass out of the cell through a pipe 10 to a blower 11 where they are somewhat compressed and forced through the gas absorber 12 through which a counter current of sodium carbonate brine is caused to flow. This brine is drawn from the tank 5 and fed through a pipe 32 including a pump 33 which feds the brine into a head tank 34 from which it flows by gravity to the absorber 12. In the absorber 12, the counter current of sodium carbonate brine combines with the carbon dioxide gas forming sodium bicarbonate crystals which are withdrawn with the liquor from the base of the absorber 12 and passed to a filter wheel 35. The recovered salts are dried in any suitable manner after they are discharged from the filter wheel and constitute one end product, namely, sodium bicarbonate. The filtrate or mother liquor from the filter wheel 35 is collected under a vacuum maintained in a receiver 36 by a vacuum pump 37. The filtrate is removed from the receiver 36 by a pump 38 and returned thereby to the brine tank 5.

The brine for flushing the absorber 12 is withdrawn from the brine tank 5 by the pump 33 which lifts the brine to the head tank 34 feeding the absorber 12.

The oxygen gas is removed from the top of the absorber or scrubber 12 by a blower 39 and passed through a pipe 40 through a suitable drier 41, wherein the drying may be accomplished through the use of sulphuric acid or by refrigeration or otherwise. From the drier 41, the oxygen gas is preferably fed to an absorber 42 of any suitable form.

Metallic sodium is fed from the hot well 23 by a pump 43 to the absorber 42, wherein it is combined with the oxygen gas therein to form sodium peroxide (Na_2O_2) which constitutes a third end product of the process. If desired, I may pass this end product through additional steps to obtain hydrogen peroxide (H_2O_2).

In practice, it is found that if all of the oxygen resulting from the electrolysis is combined with the sodium in the absorber 42 that I can utilize about one half of the metallic sodium produced by the process. If it is desired to convert all of the metallic sodium produced into sodium peroxide, I may augment the oxygen by introducing air through an inlet 44 to produce sufficient oxygen to combine with all of the sodium in which event all of the sodium produced in the hot well 23 may be fed to the absorber 42 for the making of sodium peroxide.

During the operation of the apparatus, considerable temperature may be present in the scrubber 12 and this temperature may be lowered to the desired operating temperatures by an enclosed coil 45 through which the feed water or brine for the tank 5 is fed. This feed water or brine serves to carry out the cooling function and at the same time the heat exchanged serves to maintain the desired temperature in the tank 5 and to provide feed water for the condenser 25.

During the condensation of the mercury in the heat exchanger 25, certain uncondensed gases are produced. These gases are fed out to a pipe 46 to a second condenser 47, the condensate from which is fed to the washer 28.

In carrying out the process of this invention, it is entirely practical with the proper disposition of electrolytic cells 1, still 15 and condenser 25 in relation to each other, to provide that all of the liquid mercury will flow by gravity to its destination. The necessary elevation of the mercury is accomplished by the mercury vapor from the still to the condenser and from this point, the mercury flows back to the cell by gravity and by gravity from the cell to the still.

Attention is also directed to the efficient manner in which electrical short circuiting of the system is precluded through the interposition of nonconductive liquid seals in the mercury inlet and amalgam outlet of the cell. I have referred to the use of valves embodying reciprocating pistons for controlling this inlet and outlet and have stated that these pistons may be controlled by crank disks which are synchronized. In practice, I drive the shafts of both of these crank disks in synchronism, generally by the motor, operating a variable speed transmission and I control the motor electrically from a pressure regulator 48 included in the steam outlet 27, so that by regulating the pressure of the steam, I can regulate the flow of mercury and amalgam from the cell. At the same time, I preferably associate with the mercury vapor pipe 24, a thermostatic regulator 49 and utilize this regulator to regulate an electric controlling circuit for controlling the fuel inlet valve 51 to the still 15. By these two controls, the feed of mercury to the still, the production of the resulting amalgam, and the breaking up of said amalgam are so coordinated as to produce an automatic control of the system.

In the utilization of the heat in the mercury vapors from the denuder still, I do not wish to limit myself to simple condensation of these vapors as described, but wish to point out that a turbine may be interposed in the vapor line 24 between the still 15 and the condenser 25 for the purpose of generating power and more completely utilizing the energy in the mercury vapors. The mercury vapors discharged from the turbine would be condensed in a suitable condenser recovering further heat from the mercury vapors which may be used to generate steam for additional power or other application. It is important to note that a balanced heat and power cycle may be obtained whereby the energy for electrolysis, auxiliaries and processing may be derived entirely from the heat input to the denuder still 15.

In the operation of the device, the feed of the mercury intermittently as described by the pulsating pump valves at regular intervals causes the surface of the cathode to be rippled frequently. This together with the frequent removal of the amalgam mixture from the cell assists in the diffusion of the amalgam throughout the mercury cathode and prevents prolonged exposure of the metallic sodium to contact with the water in the electrolyte, thus decreasing the liability to hydration of the amalgam and the formation of caustic soda. The mercury oil displacement is accomplished in a manner that will preclude the admission of oil into the cell or into the mercury or amalgam mixture conduits.

It is found in practice that the carbon dioxide and oxygen gases evolved in the cell are of an exceptionally high quality which give end products of high character.

Experience has shown that the process of this invention and the apparatus which I have described operates with high efficiency and produces an extremely high yield as compared to prior methods and processes. In fact, the yield is unusually close to calculated yields of theoretical efficiency and the resulting products are of high character. The electrical energy efficiency of the present process is important as power is one of the highest elements of cost in production.

The present invention is of high efficiency in this connection. Under prior practice due to numerous unavoidable conditions, decomposition efficiency with fused or molten electrolytes ranges from 40% to a possible maximum of 60%. The electric current accomplishing the decomposition is propelled through the cells through the electromotive force or the voltage difference between the two poles of the cell. The theoretical voltage required for the decomposition of an electrolyte is contingent upon so many factors, such as density of solution, temperature of solution and the physical and chemical character of the poles, that it is extremely difficult to express voltage in terms of efficiency without entering into a long discussion of the physical and chemical facts involved; however, practical operation discloses that the average voltage efficiency in a fused electrolyte cell varies from 30% to 50%. It follows that the energy efficiency in such cells may range from 12% to 30%. With the aqueous sodium carbonate electrolyte used in my cell, as previously described, the decomposition efficiency is about 95% and the voltage efficiency is from 45% to 50%, which means that the current efficiency will range between 42% and 47½%, with an average energy efficiency of about 45%, which is about 50% better than has been previously revealed for the commercial operation of cells for the production of metallic sodium.

An important step in the process of this invention is manifestly that which consists in the heating of the amalgam to break up the amalgam and cause the sodium to form a stratum which permits it to be drawn off in pure or substantially pure metallic form through a tail pipe which of itself or in conjunction with the hot well 23 is of sufficient height to balance the pressure in the still. This decanting operation is extremely simple and efficient and results in a superior product.

In the foregoing detailed description of the invention, I have chosen to describe the process and apparatus herein employed particularly in connection with a carbonate and more particularly sodium carbonate.

I prefer in practicing the present process to employ a carbonate of an alkali metal for the reasons hereinbefore stated, and more particularly because of the deleterious effects of the chlorides upon the cell and other apparatus employed in the electrolysis of such chlorides. I wish it understood, however, that the example given, viz., treatment of sodium carbonate to produce the end products described, is, for the purpose of illustration only, and that I may employ in carrying out the process of this invention halides, sulfates nitrates and oxides of an alkali metal or alkali earth metals without departing from the invention. Such procedure will, of course, produce different end products than those specifically described, but will be understood by those skilled in the art from the foregoing detailed description.

Furthermore in the employment of such other compounds to be operated upon, slight variations are necessary in the apparatus employed. For example, while a still may be conveniently used, as stated, for the decomposition of the sodium amalgam, it is desirable in connection with certain other amalgams to employ an electric furnace or some other convenient form of vaporizer. Similarly, in the treatment of the chlorides that portion of the apparatus hereinbefore described as having to do with the production of a sodium bicarbonate will of course be dispensed with, but the peroxide may be formed by using air instead of drawing on the supply of oxygen from the absorber as hereinbefore set forth.

Furthermore, I have specifically referred to the treatment of salts of alkali metals, but I wish it understood that the present invention is useful in connection with the salts of metals of the alkali earths.

For the reasons given, I wish it understood that the detailed description hereinbefore set forth is for the purpose of illustration only and that the invention is to be understood as fully commensurate with the appended claims.

By "controlled conditions", as used in the appended claims, I mean appropriate control of pressure and temperature with relation to the alkali metal under treatment.

Having thus fully described the invention, what I claim as new and desire to secure by Letters Patent is:

1. The method of recovering alkali metals and metals of alkali earths, which consists in electrolytically decomposing a salt solution thereof in the presence of mercury to produce an amalgam, vaporizing the mercury content of the resulting amalgam under controlled conditions to leave the residual alkali metal in substantially pure state, and returning the recovered mercury to the electrolytic step of the method.

2. The method of recovering alkali metals and metals of alkali earths, which consists in electrolytically decomposing a salt solution thereof in the presence of mercury to produce an amalgam, oxygen and another gas resulting from the particular anion of the salt, vaporizing the mercury content of the resulting amalgam to leave the residual metal, and combining metal thus recovered with at least one of said gases to form a compound thereof.

3. The method which consists in electrolytically decomposing a solution of a salt of an alkali metal or metal of an alkali earth in the presence of mercury to form an amalgam, oxygen and another gas resulting from the particular anion of the salt, vaporizing the mercury content of the resulting amalgam to leave the residual alkali metal, and combining alkali metal thus recovered with said oxygen gas to form a compound thereof.

4. The method which consists in electrolyzing a salt solution of an alkali metal or metal of an alkali earth in the presence of mercury to form an amalgam, heating said amalgam under controlled conditions to a temperature sufficient to volatilize the mercury content thereof but insufficient to volatilize the alkali metal content thereof, for the purpose of decomposing the amalgam through vaporization of the mercury to free the alkali metal in substantially pure state therefrom, removing such alkali metal from the zone of decomposition, and returning the recovered mercury to the electrolytic step of the method.

5. The method which consists in electrolyzing a salt solution of an alkali metal or metal of an alkali earth in the presence of mercury to form an amalgam, heating said amalgam to a temperature sufficient to volatilize the mercury content thereof but under conditions of pressure insufficient to volatilize any substantial portion of the alkali metal content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom and cause the same to form a stratum in the decomposition zone, and removing the alkali metal from said stratum thus formed.

6. The method which consists in electrolyzing a salt of an alkali metal or an alkali metal earth at a temperature below the melting point of said salt and in the presence of mercury to form an amalgam, heating said amalgam to a temperature sufficient to volatilize the mercury content thereof but under conditions of pressure insufficient to volatilize any substantial portion of the alkali metal content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom and cause the same to form a stratum in the decomposition zone, and removing the alkali metal from the stratum thereof thus formed.

7. The method which consists in electrolyzing a solution of a carbonate of an alkali metal in the presence of mercury at a temperature in the neighborhood of 200° F. to form an amalgam, heating the resulting amalgam to a temperature sufficient to volatilize the mercury content thereof, but insufficient to appreciably volatilize the alkali metal content thereof for the purpose of decomposing the amalgam to free the alkali metal therefrom, and removing the alkali metal thus formed.

8. The method which consists in electrolyzing

a solution of a carbonate of an alkali metal in the presence of mercury to form an amalgam and to evolve oxygen and carbon dioxide gases, heating the resulting amalgam to a temperature sufficient to volatilize the mercury content thereof but insufficient to volatilize the alkali metal content thereof for the purpose of decomposing the amalgam to free the alkali metal therefrom, removing the alkali metal thus formed, and subjecting at least a portion of such alkali metal to the action of said oxygen gas to provide a desired end product.

9. The method which consists in electrolyzing a solution of sodium carbonate in the presence of mercury to form a sodium amalgam, thereafter heating the amalgam to a sufficient temperature to volatilize the mercury content thereof but under conditions of pressure insufficient to volatilize any substantial portion of the alkali metal content thereof for the purpose of decomposing the amalgam to free the sodium therefrom and cause the same to form a stratum, and removing the metallic sodium from the stratum thus formed.

10. The method which consists in electrolyzing a solution of sodium carbonate in the presence of mercury to form sodium amalgam and liberate oxygen and carbon dioxide gases, thereafter heating the amalgam to a temperature sufficient to volatilize the mercury content thereof, but insufficient to volatilize the sodium content thereof, for the purpose of decomposing the amalgam to free the metallic sodium therefrom, removing the metallic sodium thus formed and subjecting at least a portion of said metallic sodium to the action of the oxygen evolved during the electrolyzing step of the process for the purpose of producing sodium peroxide.

11. The method which consists in electrolyzing a solution of sodium carbonate in the presence of mercury to form sodium amalgam and liberate oxygen and carbon dioxide gases, thereafter heating the amalgam to a temperature sufficient to volatilize the mercury content thereof, but insufficient to volatilize the sodium content thereof, for the purpose of decomposing the malgam to free the metallic sodium therefrom, removing the metallic sodium thus formed to produce substantially pure metallic sodium, and subjecting the carbon dioxide gas evolved during the electrolyzing step of the process to the action of a solution of sodium carbonate to produce sodium bicarbonate.

12. The method which consists in electrolyzing a solution of sodium carbonate in the presence of mercury to form sodium amalgam and liberate oxygen and carbon dioxide gases, thereafter heating the amalgam to a temperature sufficient to volatilize the mercury content thereof, but insufficient to volatilize the sodium content thereof, for the purpose of decomposing the amalgam to free the metallic sodium therefrom, removing the metallic sodium thus formed, subjecting at least a portion of said metallic sodium to the action of oxygen evolved during the electrolyzing step of the process for the purpose of producing sodium peroxide, and subjecting carbon dioxide evolved from the electrolyzing step of the process to the action of a solution of sodium carbonate to form sodium bicarbonate.

13. The method of separating and recovering alkali metals and metals of the alkali earths from amalgams, which consists in vaporizing the mercury therefrom, under controlled conditions and removing the mercury vapor to obtain the residual metallic alkali metal in substantially pure state.

14. The method of making an amalgam, which consists in electrolyzing a solution of a carbonate of an alkali metal in the presence of mercury and at a temperature in the neighborhood of 200° F.

15. The method which consists in electrolyzing a solution of a carbonate of an alkali metal in the presence of mercury, at a temperature in the neighborhood of 200° F., to form an amalgam, and thereafter vaporizing the mercury content of the amalgam to recover the constituent alkali metal.

16. The method of recovering alkali metals and metals of alkali earths, which consists in electrolytically decomposing a solution thereof in the presence of mercury to produce an amalgam, oxygen and another gas resulting from the particular anion of the salt, thereafter recovering the alkali metal constituent of the cell effluent, and thereafter chemically combining the said oxygen gas with a portion of the recovered metal to form an end product different from the metal, gas or electrolyte employed.

17. The method which consists in electrolytically decomposing a solution of sodium carbonate in the presence of mercury to produce an amalgam and evolve oxygen and gaseous carbon dioxide, thereafter recovering the sodium from the amalgam, and thereafter chemically combining said oxygen with a portion of the sodium recovered to form sodium peroxide.

18. The method which consists in electrolytically decomposing a solution of alkali carbonate at a temperature in the neighborhood of 200° F. to liberate oxygen and carbon dioxide gases, and subjecting the carbon dioxide gas evolved during the said decomposition step to the action of a solution of an alkali carbonate to produce an alkali bicarbonate.

19. The method which consists in electrolytically decomposing a solution of sodium carbonate in the presence of mercury to produce an amalgam and evolve oxygen and gaseous carbon dioxide, thereafter recovering the sodium from the amalgam, chemically combining said oxygen with a portion of the sodium recovered to form sodium peroxide, and chemically combining said carbon dioxide with a solution of sodium carbonate to produce sodium bicarbonate.

20. The method which consists in electrolyzing a solution of a salt of an alkali metal or alkali metal earth in the presence of a mercury cathode to form an amalgam, intermittently drawing off the amalgam from the cathode, heating said drawn off amalgam to a temperature sufficient to volatilize the mercury content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom, removing the alkali metal thus formed, and intermittently replenishing the mercury cathode in amounts to maintain a substantially constant cathode volume during the electrolysis step of the process.

21. The method which consists in electrolyzing a solution of a salt of an alkali metal or alkali metal earth in the presence of a mercury cathode to form an amalgam, intermittently drawing off the amalgam from the cathode through a dielectric seal, heating said drawn off amalgam to a temperature sufficient to volatilize the mercury content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom, removing the alkali metal thus formed, and intermittently replenishing the mercury cathode

through a di-electric seal in amounts to maintain a substantially constant cathode volume during the electrolysis step of the process.

22 The method which consists in electrolyzing a solution of a salt of an alkali metal or alkali metal earth in the presence of a mercury cathode to form an amalgam, intermittently drawing off the amalgam from the cathode through a di-electric seal, heating said drawn off amalgam to a temperature sufficient to volatilize the mercury content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom, removing the alkali metal thus formed, and intermittently replenishing the mercury cathode through a di-electric seal in amounts to maintain a substantially constant cathode volume during the electrolysis step of the process, and replenishing the electrolyte intermittently through a di-electric seal.

23. The method which consists in electrolyzing a solution of a salt of an alkali metal or alkali metal earth in the presence of a mercury cathode to form an amalgam, intermittently drawing off the amalgam from the cathode through .a di-electric seal, heating said drawn off amalgam to a temperature sufficient to volatilize the mercury content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom, condensing the mercury vapors evolved during the heating step of the process, removing the alkali metal thus formed, and intermittently replenishing the mercury cathode through a di-electric seal in amounts to maintain a substantially constant cathode volume during the electrolysis step of the process, and controlling the feed of mercury and amalgam to and from the cathode by the pressure of the mercury condensing medium.

24. The method which consists in electrolyzing a solution of a salt of an alkali metal or alkali metal earth in the presence of a mercury cathode to form an amalgam, intermittently drawing off the amalgam from the cathode through a di-electric seal, heating said drawn off amalgam to a temperature sufficient to volatilize the mercury content thereof, for the purpose of decomposing the amalgam to free the alkali metal therefrom, condensing the mercury vapors evolved during the heating step of the process, removing the alkali metal thus formed, intermittently replenishing the mercury cathode through a di-electric seal in amounts to maintain a substantially constant cathode volume during the electrolysis step of the process, controlling the feed of mercury and amalgam to and from the cathode by the pressure of the mercury condensing medium, and controlling the temperature of said heating step by the temperature of the mercury vapors evolved during such step.

25. The method of recovering metallic sodium from a sodium salt solution which consists in electrolytically decomposing such sodium salt solution in the presence of mercury to produce an amalgam, vaporizing the resulting amalgam under controlled conditions to leave the residual metallic sodium in substantially pure state and returning the recovered mercury to the electrolytic step of the method.

SETH A. MOULTON.