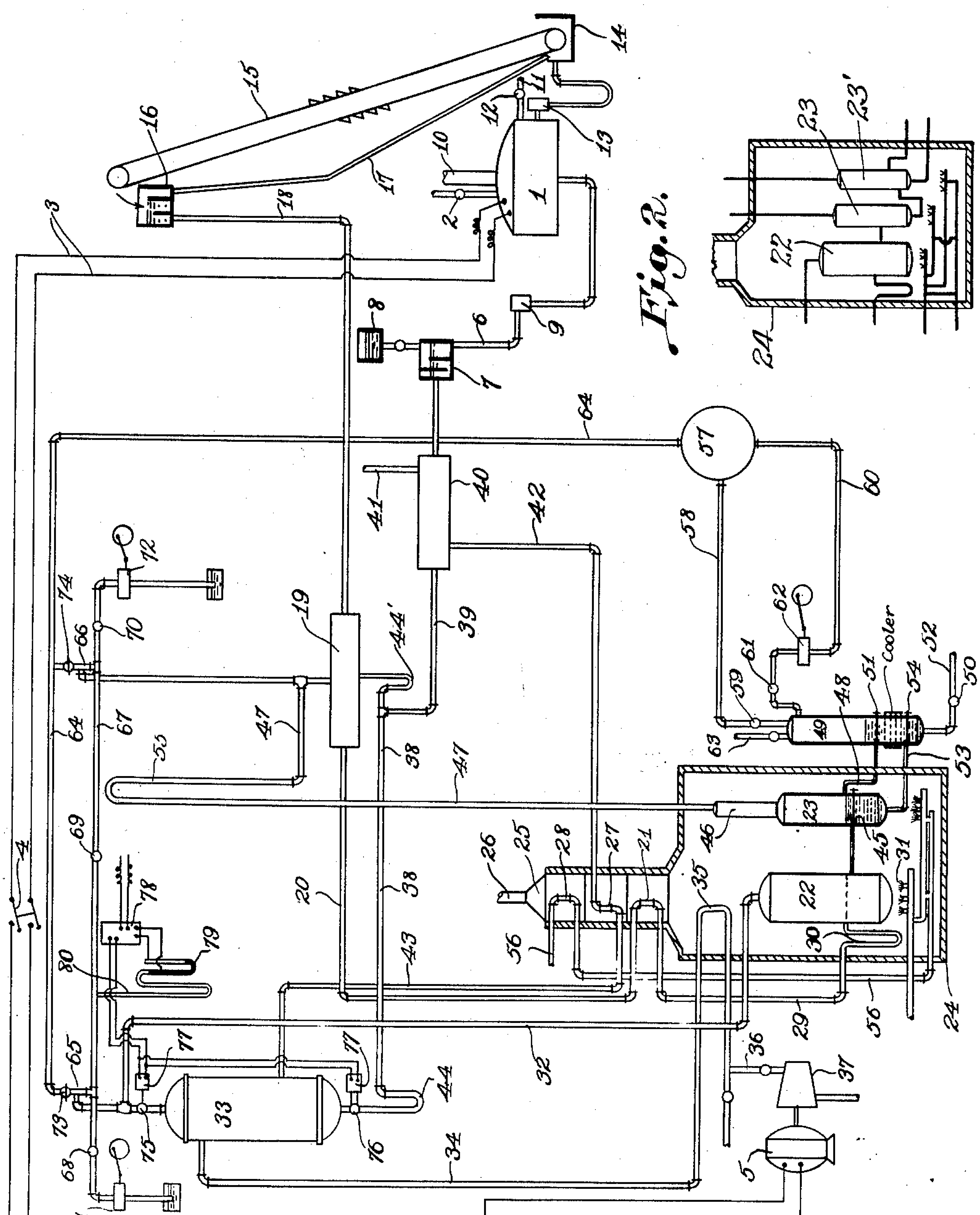
June 5, 1934. P. F. CRAHAN ET AL 1,961,135

METHOD OF AND APPARATUS FOR EXTRACTING METALS FROM AMALGAMS

Filed June 9, 1933



INVENTOR.
Peter F. Crahan
Seth A. Moulton
Gordon E. Seavoy

BY [signature]

ATTORNEY.

Patented June 5, 1934

1,961,135

UNITED STATES PATENT OFFICE

1,961,135

METHOD OF AND APPARATUS FOR EXTRACTING METALS FROM AMALGAMS

Peter F. Crahan, New York, N. Y., and Seth A. Moulton, Los Angeles, Calif., and Gordon E. Seavoy, Kew Gardens, N. Y.; said Moulton and said Seavoy assignors to said Crahan

Application June 9, 1933, Serial No. 675,056

19 Claims. (Cl. 75—17)

This invention is a method of extracting metals from amalgams and includes novel apparatus by means of which the method may be commercially performed. More particularly, the invention is directed to extracting alkali metals and metals of alkali earths from amalgams preferably formed through the electrolysis of salts of such metals in the presence of a mercury cathode.

Alkali metals and metals of alkali earths can be recovered by the electrolysis of a salt of a metal in a cell employing a mercury cathode, to produce an amalgam, this step being followed by heat treatment of the amalgam for the purpose of disassociating the mercury of the amalgam from the alkali metal content thereof. The mercury may be volatilized and led off as a vapor leaving the alkali metal as a residue.

A method and apparatus for accomplishing this result is described and claimed in an application filed by Seth A. Moulton on January 14, 1933, Serial No. 651,704. In accordance with the method of said application which, so far as we know, contains the first disclosure of a practical manner of accomplishing this result, the amalgam is heat treated at a temperature sufficiently high to effect the disassociation referred to. Said method and apparatus have been practically operated and have produced substantially pure alkali metal, but certain problems have arisen which it is the purpose of the present invention to overcome. For example, it has been found that, if the concentration of the alkali metal in the cell effluent is too high, the process cannot be practically carried out. It therefore becomes desirable, in the interest of a continuous commercial process, to work with relatively low concentrations. This means that the cell effluent will contain not only an amalgam, but an appreciable amount of free mercury which acts as a vehicle for the amalgam in the subsequent steps of the process. If this entire effluent is passed through a single heat treatment, the temperature must be sufficiently high to effect a disassociation of the amalgam and the temperature is considerably in excess of that required for the vaporization of pure mercury.

The disadvantages of this procedure will be hereinafter more fully enumerated, but suffice it here to say that the primary object of the present invention is to eliminate the necessity of treating the entire cell effluent at maximum temperatures and to act upon the cell effluent by successive steps or stages of appropriate temperatures in each stage, so as to first remove the free mercury at a temperature appropriate to this operation, and thereafter disassociate the constituents of the amalgam at an appropriate temperature or temperatures to remove the mercury thereof and leave the alkali metal in substantially pure state as a residue.

The present invention is the result of protracted study and experimentation on our part which has shown that complete and practical separation of two metals, such, as mercury and an alkali metal, should, for best commercial purposes, be accomplished in two or more definite stages, for, as stated, there are at least two unit processes or steps involved in the segregation of these metals. First, simple distillation or vaporization of free excess mercury in the cell effluent in, e. g., one still, and, second, the decomposition of the residual amalgam of the two metals in, e. g., one or more additional stills, to effect the ultimate separation of said metals.

While the steps of recovery of the alkali metal from the cell effluent constitute the primary object of this invention, the invention embodies further objects which will hereinafter more fully appear, but which have to do more particularly with those method steps and apparatus which enter into a commercial system for effecting the purposes stated.

For example, an important feature of this invention is inherent in the employment of a relatively large mass or volume of recovered alkali metal at such a temperature that amalgam, when brought in contact therewith, is flashed to effect thermal decomposition thereof, as hereinafter explained in detail.

A further important feature of this invention resides in the use of an inert fluid to facilitate drawing off of the recovered alkali metal without admission of atmospheric air into the system, and the further use of this inert fluid as a safety medium, so that in the event the vacuum is broken in the system through failure of apparatus or any other cause, the inert fluid may be fed, preferably automatically, into the system to protect the flooding of the system by air, steam or the like.

Another feature of the invention which is highly important from a commercial standpoint has to do with proper heat recovery, so that the energy in the fuel employed may be efficiently recovered and utilized to do useful work.

Another feature of the invention consists in the employment of a refluxing step functioning in conjunction with the thermal decomposition of the amalgam and serving to protect against the loss of the alkali metal, while permitting efficient vaporization of the mercury constituent of the amalgam.

Aside from the foregoing, the invention involves various safety factors which will be presently enumerated.

We are aware that in prior patents and in the literature of science, prognostications have been made with reference to the decomposition of amalgams, but these prior publications are wholly lacking in the disclosure of any commercial workable method or apparatus whereby the results accomplished by this invention can be obtained.

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

The accompanying drawing illustrates diagrammatically one form of novel apparatus for carrying out the main and sub-processes of this invention. We wish it understood, however, that the apparatus shown is illustrative, only, and other forms of apparatus may be employed without departing from the invention.

In the drawing, Figure 1 is a diagrammatic showing of the apparatus.

Figure 2 shows a modified form of one part of the apparatus.

The invention may be carried out in conjunction with various alkali metals or metals of alkaline earths, such, for example, as sodium, potassium, caesium, barium, etc., but, for the purpose of concrete illustration, we have chosen to describe sodium as the metal which it is desired to recover. The following description, therefore, will deal specifically with this metal, it being understood that the invention is not expressly limited thereto.

Furthermore, in the following detailed description, we shall set forth not only the main process of this invention, but various sub-processes or method steps which also constitute a part of the present invention, and it is therefore to be understood that some parts of this disclosure may be employed within the scope of the present invention without necessarily employing all. However, the apparatus shown in a more or less diagrammatic manner is that of a complete system susceptible to commercial use in the carrying on of the process.

In adapting the invention to the recovery of sodium, a suitable brine, such, for example, as sodium carbonate, is fed from a suitable source of supply, not shown, to one or more electrolytic cells, one of which is indicated at 1. The brine is fed through an inlet 2 provided with an appropriate interrupter, so that the brine may be fed into the cell at predetermined intervals or substantially constantly through an interrupted flow, in order that the cell may not be electrically short circuited. The cell is of the type employing a mercury cathode and appropriate current is supplied through a circuit 3, including a switchboard 4, from a suitable source of current supply indicated as a generator 5.

Mercury is adapted to be supplied to the cathode through a pipe 6, which leads from a scrubber 7, preferably fed from mercury which is passed through the process and has been recovered or from fresh mercury fed from a source of supply 8, or from both. In any event, mercury passes through pipe 6 and through an interrupter 9 into the cell to form the cathode. The interrupter 9 functions to intermittently feed the mercury in a manner to break the mercury column and thus preclude short circuiting.

The sodium carbonate fed into the cell is electrolyzed therein, the sodium combining with a portion of the mercury cathode to form an amalgam and the generated gases passing off through an outlet 10, where they may be utilized or treated in any suitable manner, forming no part of this particular invention. The brine level in the cell is meanwhile kept constant by an overflow 11 provided therein with an interrupter 12 to preclude short circuiting. The amalgam formed in the cell is led out through an interrupter 13 to a well 14. The connection between the cell and this well may be in the form of a loop in order to hold a slight vacuum in the cell if this is desired.

Amalgam from the well 14 is fed by any suitable conveyor, such, for example, as an elevator 15 to a flow box 16, which, by preference, is positioned at such elevation as to permit the method to operate in the main as a gravity flow system from this point on. It is for this reason that we employ the elevator 15, so as to obtain this single point of elevation. Another reason for employing the elevator, but more particularly a flow box in the form of a weir, is that we may also employ in connection therewith a return overflow 17, and by adjustment of the weir, the feed of amalgam through the subsequent steps of the process may be accurately controlled as to volume.

The amalgam passes from the flow box 16 through a pipe 18 to a heat exchanger 19. This heat exchanger may be of tubular character, the amalgam passing through the tubes and vapors circulating around the tubes, as hereinafter more fully explained. The vapors employed are of highly heated character and the heat exchanger in this particular step of the process is employed to raise the temperature of the amalgam, so that it passes from the exchanger 19 through pipe 20 in a heated condition. The pipe 20 leads to a heat exchanger 21 forming part of a heat economizer employing the heat generated in subsequent vaporizing and disassociation steps of the process, and in this heat exchanger 21, the temperature of the amalgam is appreciably raised, i. e., it is raised to a point where it can be economically fed to the next step of the process which constitutes, generally speaking, a vaporizing tep.

This vaporizing step is carried out first within a primary still or stills 22 and a subsequent disassociation step is carried out within a secondary still or stills 23. The respective stills may have their own individual source of heat and they may be heated to various or progressive temperatures as may be desired. However, for the purpose of concrete illustration, we have shown these several stills (two, for example) as contained within a common casing or furnace 24 having an outlet chamber 25 leading to the stack 26, and this outlet chamber is divided into a plurality of heat economizers. The heat economizer 21 has already been referred to, but, as shown, there are two additional economizers 27 and 28, which will be hereinafter more fully explained. It will be noted that the infeed 29 which leads from the economizer 21 to the primary still 22 is provided therein with a U-tube and vacuum seal 30.

We have hereinbefore referred in the flow or passage of amalgam from the cell through various apparatus, pipes, etc., to a primary still. It is essential at this point to clearly explain an important factor of this invention and one which goes to the very base of the present process.

As hereinbefore stated, the process of this invention is primarily directed to a commercial means of obtaining, for example, sodium from a brine thereof. This brine is electrolyzed and the sodium is combined with part of the mercury forming the cathode with a view to subsequently breaking up the amalgam into its mercury and sodium constituents and separating them to obtain the free sodium. Now, as a matter of fact, in order to efficiently carry on a process of this kind, it is not deemed practical to operate on a cell effluent of amalgam alone which would presuppose a total conversion of the mercury cathode into an amalgam. In other words, it would require a combination of all of the mercury in the cell with the sodium from the brine and would produce a cell effluent having a very high sodium concentration. Such an effluent would be unsatisfactory for use in the present process for many important reasons. In the first place, such an effluent would be practically a solid at ordinary temperatures and could not be subjected to a flow.

Another disadvantage of employing such a high sodium concentration in the cell is that, if used, counter reactions with the electrolyte would occur with consequent appreciable reduction in the efficiency of the cell. There are additional reasons why the course stated is impractical, but those referred to are sufficient to illustrate.

It is of course desirable to retain the fluidity of the cell effluent in order that it may be properly handled. This can be most efficiently accomplished by lowering the percentage concentration of sodium allowed to combine with the cathode of a cell during the electrolytic step.

In carrying out this invention, we can operate of course within relatively wide limits, so far as actual concentration is concerned, depending upon several factors. However, for the purpose of concrete example, and without in anywise intending to limit the invention to specific precentages, it has been found that a sodium concentration of 0.15% by weight in the cell effluent will give highly satisfactory and efficient results.

When it is borne in mind that the cell effluent contains, in the example set forth, 0.15% sodium, it becomes apparent that this sodium is combined with only a portion of the mercury cathode to form an amalgam, and that with this amalgam, there are relatively large amounts of free mercury which, as a matter of fact, act as a vehicle for the amalgam in passing through the steps of the process thus far described. Accordingly, the effluent which leaves the cell embodies a metallic mercury as a vehicle for entrained sodium amalgam.

The present invention provides a relatively simple and economical method and means, whereby it is made possible to obtain from this effluent the alkali metal constituent in a substantially pure state.

In practice, the difficulty of breaking up the effluent into its constituent parts for the purpose of recovery of the mercury and the alkali metal is due largely to the different conditions which are best adapted for recovery of the particular constituents and for disassociation of the mercury from the sodium in the amalgam. For example, relatively pure mercury will volatilize at a lower temperature than is required to disassociate the constituents of the amalgam and it has been found that if the amalgam with the mercury vehicle is placed in a single still and subjected to sufficient temperatures to totally disassociate the mercury in the amalgam, the entire cell effluent must be subjected to the maximum temperatures. This produces undesirable wear and tear on the apparatus and conditions very difficult of control in order to obtain a pure product. It is not impossible to obtain the pure product by the former process, but by the present invention provision is made for a more economical, simple and efficient method of obtaining this result and with a higher safety factor.

According to the present invention, therefore, we effect a disassociation, both physical and chemical, by a succession of steps or stages. In the first stage, the effluent from the cell, preferably preheated in the interest of economy in the heat exchangers 19 and 20, is fed into the primary still 22 heated in any appropriate manner as by a burner 31 within the furnace 24. These burners are shown as fed with air through a pipe 56 in which is included the heater 28 hereinbefore referred to.

Sufficient heat is supplied in this primary still 22 to the entire cell effluent to volatilize the free mercury vehicle thereof. This is accomplished under a partial vacuum, i. e., under a pressure lower than atmospheric pressure. The amount of vacuum employed may vary within appreciable limits without departing from this invention, but, by way of illustrative example, it may be stated that the process has been efficiently worked with a temperature and pressure wherein the vapors leave the still 22 at approximately 525° F. and 25″ vacuum (referred to a 30″ barometer). Thus, in the primary still 22, the free mercury vehicle is separated from the amalgam leaving an amalgam as a residue. It is possible with variation of pressures and temperatures to partially decompose the amalgam, so as to free some mercury therefrom, but, in the preferred method of carrying out the process, this is negligible, if it occurs at all, in its effect on the conditions of operation in the still 22, it being the function of this still primarily to remove the free mercury vehicle.

In the preferred form of the invention, the operation of separating the mercury vehicle is accomplished in a single still, although it is possible without departing from this invention, to accomplish this result by the employment of a plurality of stills 22 operable in series or parallel. In any event, the mercury vapors from the primary still or stills pass out through a duct 32 to a condenser 33 which may function, in effect, as a steam boiler, a part of the heat from the mercury vapors during condensation being utilized to convert water in the condenser 33 into saturated steam, which is fed through a pipe 34 through a superheater 35 in the furnace, whence it passes through pipe 36 to a turbine 37. The turbine may be used for any power purpose, but, is illustrated as coupled to a generator 5 which supplies electrical current to the cell 1. The condensed mercury leaves condenser 33 through a pipe 38, having therein a vacuum U tube 44, and passes through a pipe 39 to a heat exchanger 40 wherein it serves to partially heat the feed water for the condenser 33. The feed water is fed through pipe 41 to heat exchanger 40, thence through pipe 42 to the heat exchanger 27 of the economizer associated with the furnace, and thence through pipe 43 to the primary condenser 33 to be there converted into steam for use in the turbine. If desired, the steam from the condenser may be used for other purposes than to generate power without departing from this invention.

After the free mercury has been removed or separated from the amalgam in the primary still which constitutes, in effect, the first stage of separation, the resulting residue, constituting the amalgam, is fed through a valved inlet into the second stage. This second stage may constitute a single still, the temperature and pressure of which are maintained appropriate to break up the amalgam to disassociate the mercury thereof from the sodium and volatilize the former.

In practice, this stage may be accomplished in a single step employing a single still or in a plurality of steps employing a succession of stills heated to progressively higher temperatures. Figure 2 of the drawing shows two of such stills 23—23′ connected in series. For the purpose of illustration in Figure 1, however, we have chosen to show the simplest form embodying a single still in the second stage. This still is designated 23 and the inlet from the primary still is controlled by a valve 45. A body of sodium in molten state is maintained within this still with its surface level above the valve 45 and the amalgam is fed from the primary still through this valve into contact with the body of molten sodium maintained at such temperature that it will preferably immediately flash the amalgam, i. e., effect a decomposition thereof on introduction and contact with the sodium. As a result, the mercury constituent of the amalgam will become immediately vaporized, will rise to the surface of the molten sodium and leave the top of the still.

In practice, appropriate temperatures and pressures are employed to effect an efficient disassociation of the elements in this step. In practically carrying out this step, we have utilized, for the purpose of illustration, and without in anywise tending to limit this invention, a temperature and pressure of approximately 1050° F. and 29¼″ vacuum, respectively (referred to a 30″ barometer).

When thus practised, however, more or less sodium is volatilized and for the recovery of this sodium, a refluxing tower 46 is associated with the top of the still 23. The temperature of this tower is such that it will condense and reflux the sodium and permit the mercury vapor to pass out through an outlet pipe 47. With this method of procedure, it is found that sodium in a practically pure state will accumulate in the still, and may be drawn therefrom as hereinafter explained. If the valve 45 is not accurately set, this is not series, for if a greater amount of amalgam is fed into the still than can be immediately flashed, it will fall into the bottom of the still and into the zone of greatest heat therein to there be disassociated and the mercury therein volatilized.

In carrying out the process, as described, the surface level of the molten sodium in the still 23 is maintained above the amalgam inlet and the accumulated sodium in excess of that necessary for this purpose is drawn off from time to time as may be desired through a pipe 48 into a receiver 49. The pipe 48 leaves the still at an elevation above the valve 45, so as to at all times maintain a minimum level above this valve.

Practically pure sodium enters the receiver 49 and may be drawn therefrom, as desired, through a draw off valve 50. The valve 51 controls the drawing off of the sodium from the still 23 into the receiver 49 and in practice this is preferably intermittent, in order that the sodium after entering the receiver 49 may be allowed to cool before drawing the sodium therefrom through the valve 50 to final disposition through a valved pipe 52. It will be noted that pipe 53 leads from the bottom of the still 23. This is a drain pipe and is controlled by a valve 54. The drawing off of the sodium from the receiver 49 is preferably accomplished without venting to the atmosphere, as will be presently explained.

The mercury vapors evolved in the still 23 pass through the pipe 47 to the heat exchanger 19, wherein the heat thereof is given up at least in part to the mercury amalgam flowing through the pipe 20 to the still of the first stage and from the heat exchanger 19, the mercury condensed from vapors flowing into the condenser through pipe 47 passes through U-tube 44′ to meet the condensed mercury from the primary condenser 33 flowing through the pipe 38 and to pass therewith through pipe 39 to heat exchanger 40 where further heat is given up to heat the feed water, as hereinbefore described. The resulting cooled mercury is fed through the scrubber 7 to be reintroduced, through the pipe 6 and interrupter 9, into the cell.

In carrying out a process of this kind, great care should be taken to provide the greatest possible safety factors and to guard in every conceivable way against accidents which might occur from failure of apparatus. For example, in the system as described, the mercury vapors coming from the second stage are utilized to heat the mercury-amalgam mixture passing to the first stage. If, for example, the heat exchanger employed for this purpose should fail for one reason or another and allow amalgam or mercury in appreciable quantities to flow back through the pipe 47 into the still 23, this mercury would be brought immediately into contact with a relatively large body of melted sodium at high temperature with probably disastrous results. Consequently, the present invention includes the supplementary invention of providing a riser seal 55 included in the pipe 47. This riser seal is shown in the form of an inverted U-tube rising to a height above the mercury head afforded by the flow box 16, plus a sufficient elevation to compensate for the vacuum in the system. With this safety adjunct, it is apparent that that portion of the pipe 47 between the riser seal 55 and the seal 23, is absolutely safeguarded against retrograde flow of mercury. This is a novel and important safety feature in the system as shown.

In carrying out the system of this invention, various pressures, generally below atmospheric pressure, are utilized throughout the system, as hereinbefore explained. These pressures must be taken into consideration in the operation of the apparatus from various angles. For example, it is necessary to break the vacuum at the receiver 49 to draw off the final sodium product. Similarly, it is highly desirable in a system of this kind to protect the apparatus against the influx of air in the event of mechanical failure of any part. For these purposes and others, the present invention contemplates the employment of an inert fluid, which is adapted to be automatically or manually, in some cases, fed into the system at the time of removal of products therefrom, or, in the event of mechanical break down.

Thus, there is shown at 57 a source of supply for an inert fluid which may, in practice, be contained in a tank. A pipe 58 leads from this tank to the top of the receiver 49 and includes a valve

59. Another pipe 60 having a valve 61 therein and including a vacuum pump 62 leads back to the tank 57. Valved pipe 63 is an emergency vent to the atmosphere. The valved pipe 63 is closed at all times. To draw off sodium from the receiver 49, the valve 61 is closed and draw off valve 50 is opened.

It will be understood of course that the valve 51 is closed and has been closed for a time to permit sodium in the receiver to cool as desired. As soon as the draw off valve 50 is opened, the sodium flows from the receiver and is displaced by the inert fluid from tank 57. After the sodium has been drawn off, draw off valve 50 is closed, valve 59 is also closed, valve 61 is opened and the vacuum pump 62 operated to pump the inert fluid from the receiver 49 back into the tank 57 in order to restore in the interior of the receiver 49 the desired pressure. Valve 61 is then closed.

Various inert fluids may be used for this and other purposes, but Argon gas gives very satisfactory results.

It will be noted also that a pipe 64 leads from the tank 57 and connects through branch pipes 65 and 66 with a pipe 67 having therein valves 68, 69 and 70. The opposite ends of the pipe 67 connect with vacuum pumps 71 and 72. Two pumps are shown in the drawing, the pump 71 being intended primarily to maintain proper pressures in the primary still 22 and on the mercury side of the condenser 33. The pump 72 on the other hand is intended to maintain a proper pressure in the secondary still 23 by way of pipe 47 and also to maintain proper pressure in the heat exchanger 19. The pumps are connected, as shown, by a pipe with valves 68, 69 and 70 to permit of variations of this arrangement if desired. Ordinarily, the valves 68 and 70 are opened, while the valve 69 is closed. In the branch pipes 65 and 66 are automatic vacuum breakers 73 and 74, which, so long as the system is functioning properly, serve to hold the predetermined vacuums. Adjacent the opposite sides of the primary condenser 33, the safety valves 75 and 76 are included in the mercury inlet and outlet pipes and these valves are respectively controlled by automatic controllers 77 included in an electric circuit with a relay 78. This relay is actuated by a mercury U-tube contacter 79 connected by a pipe 80 to the pipe 67.

Now, for the purpose of illustration, suppose, for example, that one of the tubes in the primary condenser 33 fails. If steam were permitted to pass through the system very serious results might occur. According to the apparatus shown, however, as soon as the tube fails, the vacuum will drop and the increased pressure in the pipe 80 will cause the mercury in the U-tube 79 to make electrical contact, actuating the relay 78, which, in turn, will operate the valve closers 77 to close the valves 75 and 76. At the same time instead of admitting atmospheric air into this system, the valve 73 will be free to open to admit the inert fluid. Appropriate means are provided for stopping the vacuum pump in the event of failure of any part of the system. The valve 74 acts in the same manner with respect to the secondary stage of the operation, although no control valves are necessary because of the riser seal 55.

It will be apparent from the foregoing detailed description that we can efficiently and economically extract alkali metals or metals of alkaline earths which we look upon generically as alkaline metals. In the appended claims, we have used this term "alkaline metals", as referring to either or both alkali metals or metals of alkaline earths and the claims are to be so construed.

We have hereinbefore described the use of an inert fluid as a safety factor and to facilitate the draw off of the end product of this process. This use of an inert fluid is particularly desirable in the separation and recovery of alkaline metals and more especially sodium. We are aware, however, that the employment of inert fluids to blanket portions of systems normally operated under sub-atmospheric conditions is susceptible to use in various arts other than the recovery of alkaline metals and the present invention is not limited to this particular art, so far as this safety measure is concerned.

It will of course be understood that in utilizing such an inert fluid for the purposes stated, this fluid should be maintained under at least atmospheric pressure, and in practice the pressure of the fluid is preferably somewhat higher than atmospheric pressure, in order that the flow of this fluid into the system may be rapid and efficient in the carrying out of its intended functions.

The foregoing detailed description sets forth the invention in its preferred practical form, but the invention is to be understood as fully commensurate with the appended claims.

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

1. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in heating the mixture to a temperature to distill off the mercury from the mixture and leave the amalgam as a residue, and thereafter thermally decomposing the amalgam at a higher temperature and removing the resulting mercury vapor to leave the constituent alkaline metal as a residue.

2. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury from the mixture in a suitable vessel, thereupon transferring the remaining amalgam to another and different vessel, and thermally decomposing the amalgam in the second vessel and removing the mercury vapors produced in the second vessel to leave the constituent alkaline metal as a residue.

3. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the excess mercury in one unit process, then thermally decomposing the amalgam in a second unit process to leave the constituent alkaline metal as a residue.

4. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the excess mercury in one unit process, then thermally decomposing the amalgam in a second unit process to leave the constituent alkaline metal as a residue, and refluxing the vapors of the constituent alkaline metal which may be evolved during the second unit process.

5. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in heating the mixture in a suitable still to distill off the mercury thereof, then transferring the remaining amalgam to a second still, decomposing the

amalgam in said latter still, and vaporizing off the mercury content thereof to leave the constituent alkaline metal as a residue.

6. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in heating the mixture in a suitable still to distill off the mercury thereof, then passing the remaining amalgam in series through a plurality of additional stills heated to consecutively higher temperatures for the purpose of decomposing the amalgam and distilling off the constituent mercury thereof to leave the constituent alkaline metal as an end product.

7. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in heating the mixture in a suitable still to distill off the mercury thereof, then passing the remaining amalgam in series through a plurality of additional stills heated to consecutively higher temperatures for the purpose of decomposing the amalgam and distilling off the constituent mercury thereof to leave the constituent alkaline metal as an end product, and refluxing the vapors of the alkaline metal in at least one of the latter stills.

8. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury of the mixture, and thereafter decomposing the resulting amalgam by contact thereof with a body of molten alkaline metal of the same kind as the alkaline constituent of the amalgam and which metal is maintained at a temperature above the decomposition temperature of the amalgam, and removing the mercury vapors.

9. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury of the mixture, and thereafter decomposing the resulting amalgam by contact thereof with a body of molten alkaline metal of the same kind as the alkaline constituent of the amalgam and which metal is maintained at a temperature above the decomposition temperature of the amalgam, removing the mercury vapors, and refluxing the alkaline metal vapors evolved during the decomposition of the amalgam.

10. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury of the mixture, thereafter decomposing the resulting amalgam by contact thereof with a body of molten alkaline metal of the same kind as the alkaline constituent of the amalgam and which metal is maintained at a temperature above the decomposition temperature of the amalgam, removing the mercury vapors, refluxing the alkaline metal vapors evolved during the decomposition of the amalgam, and adding the refluxed alkaline metal as well as the unvaporized alkaline metal constituent of the amalgam to the said molten metal body.

11. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury from the mixture in a suitable vessel, thereupon transferring the remaining amalgam to another and different vessel, and thermally decomposing the amalgam in the second vessel and removing the mercury vapors produced in the second vessel to leave the constituent alkaline metal as a residue, drawing off quantities of the alkaline metal residue from time to time into a sealed chamber, drawing off quantities of said metal from said sealed chamber from time to time, and venting said sealed chamber with an inert fluid during the drawing off of the metal from the latter.

12. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in distilling off the mercury from the mixture in a suitable vessel, thereupon transferring the remaining amalgam to another and different vessel, and thermally decomposing the amalgam in the second vessel and removing the mercury vapors produced in the second vessel to leave the constituent alkaline metal as a residue, drawing off quantities of the alkaline metal residue from time to time into a sealed chamber, drawing off quantities of said metal from said sealed chamber from time to time, and venting said sealed chamber with an inert fluid during the drawing off of the metal from the latter, and recovering excess inert fluid from said chamber at the conclusion of each draw off operation.

13. The method of extracting alkaline metals from a mixture of mercury and an amalgam of an alkaline metal, which consists in feeding under hydrostatic pressure a mixture of mercury and an alkaline metal amalgam to a suitable still, decomposing the amalgam in said still to evolve mercury vapors, passing said mercury vapors into heat exchangeable relation with the mixture of mercury and amalgam on the way to the still, and causing said mercury vapors to be fed into said heat exchanging relation from a point elevated above the elevation of the hydrostatic head of the mixture of mercury and amalgam to be heated thereby.

14. The method of decomposing an alkaline metal amalgam, which consists in introducing the same into a still, applying heat thereto for the purpose of decomposing the amalgam to vaporize the mercury constituent and leave the alkaline metal as a residue, carrying on this decomposition step under sub-atmospheric pressure, maintaining an inert fluid under a pressure at least equal to that of the atmosphere, restraining the entrance of inert fluid into the still, so long as the pressure in said still is below a predetermined maximum, and admitting said inert fluid into the still when the pressure therein rises above said predetermined maximum.

15. The method of operating a closed system which consists in maintaining at least a portion of such system at sub-atmospheric pressure, maintaining a body of inert fluid at a pressure at least as high as atmospheric pressure, restraining the entrance of inert fluid into the sub-atmospheric pressure portions of the system, so long as the pressure in said portion of the system is below a predetermined maximum, and admitting said inert fluid into the system when the pressure in the sub-atmospheric portion of the system rises above said predetermined maximum.

16. In an apparatus of the character described, a cell for producing an effluent mixture of mercury and an amalgam of an alkaline metal, a primary still, means for feeding said mixture from the cell to the primary still, means for heating the mixture in the still to distill off the free mercury thereof, a secondary still, means for maintaining a body of alkaline metal in the secondary still, means for feeding predetermined quantities of amalgam from the primary still and intro-

ducing it into the secondary still below the level of the alkaline metal body therein, and means for maintaining said alkaline metal at a temperature to thermally decompose the amalgam upon contact with said body and to distill off the mercury constituent of said amalgam.

17. In an apparatus of the character described, a cell for producing an effluent mixture of mercury and an amalgam of an alkaline metal, a primary still, means for feeding said mixture from the cell to the primary still, means for heating the mixture in the still to distill off the free mercury thereof, a secondary still, means for maintaining a body of alkaline metal in the secondary still, means for feeding predetermined quantities of amalgam from the primary still and introducing it into the secondary still below the level of the alkaline metal body therein, means for maintaining said alkaline metal at a temperature to thermally decompose the amalgam upon contact with said body and to distill off the mercury constituent of said amalgam, and a refluxing tower connected to the secondary still to reflux vapors of alkaline metal evolved in the secondary still.

18. In an apparatus of the character described, a cell for producing an effluent mixture of mercury and an amalgam of an alkaline metal, a primary still, means for feeding said mixture from the cell to the primary still, means for heating the mixture in the still to distill off the free mercury thereof, a secondary still, means for maintaining a body of alkaline metal in the secondary still, means for feeding predetermined quantities of amalgam from the primary still and introducing it into the secondary still below the level of the alkaline metal body therein, means for maintaining said alkaline metal at a temperature to thermally decompose the amalgam upon contact with said body and to distill off the mercury constituent of said amalgam, a refluxing tower connected to the secondary still to reflux vapors of alkaline metal evolved in the secondary still, a heat exchanger interposed between the cell and the primary still and through which said mixture is adapted to pass, and a conduit for feeding mercury vapors from one of said stills through said heat exchanger to permit heating of the mixture by said mercury vapors with accompanying condensation of said vapors, said conduit having therein a riser seal extending to an elevation exceeding that of the maximum pressure head of the cell effluent mixture.

19. The method of extracting an aklaline metal from an amalgam mixture including such alkaline metal, which consists in decomposing the amalgam mixture by contact with a body of molten alkaline metal of the same kind as the alkaline constituent of the amalgam and maintained at a temperature above the decomposition temperature of the amalgam, and removing the mercury vapors.

PETER F. CRAHAN.
SETH A. MOULTON.
GORDON E. SEAVOY.