W. L. MORRISON.

PROCESS FOR PRODUCING ALKALI METALS OR THE LIKE.

APPLICATION FILED AUG. 17, 1916.

1,265,360.

Patented May 7, 1918.

3 SHEETS—SHEET 1.

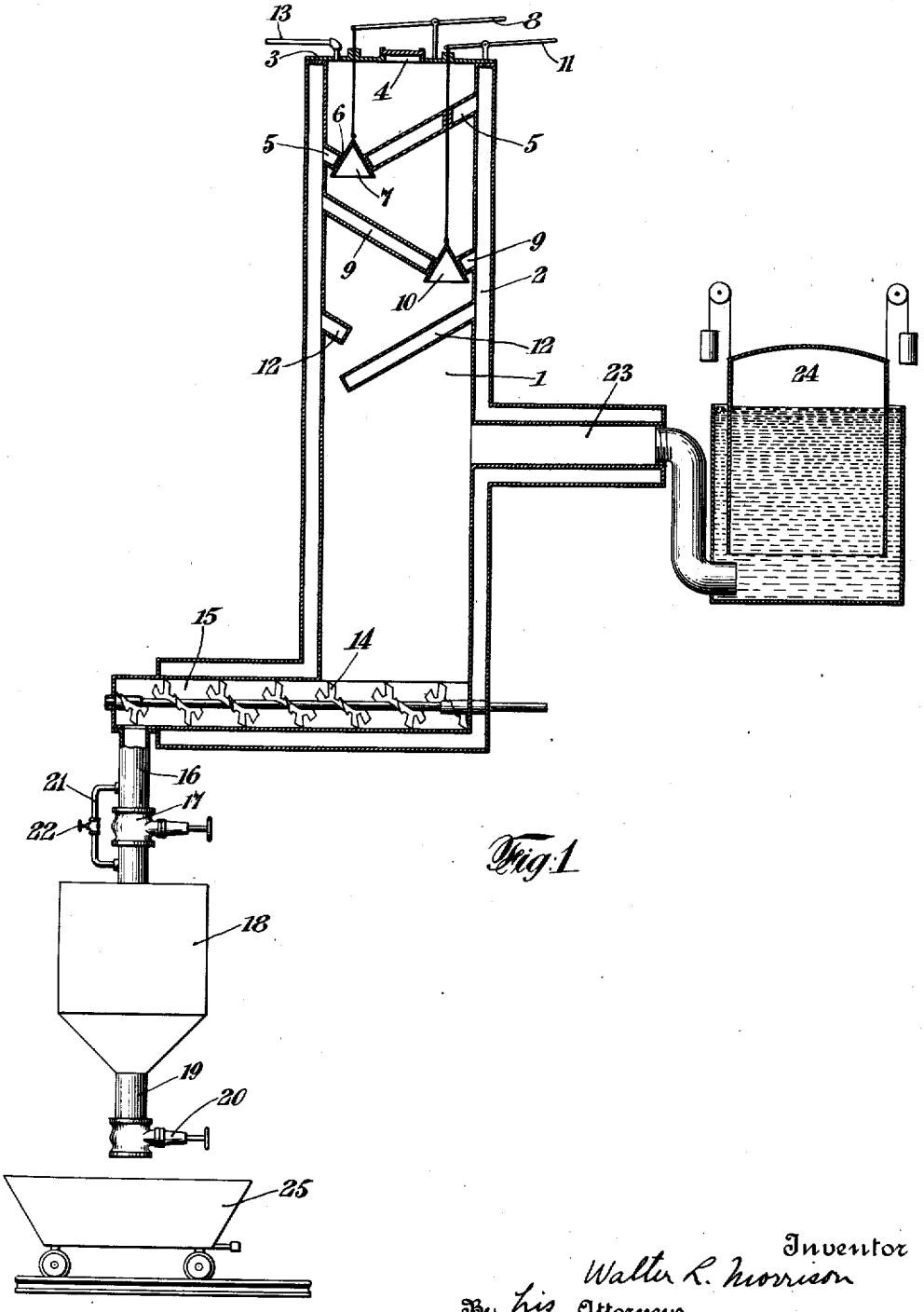


Fig. 1

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Fig. 2

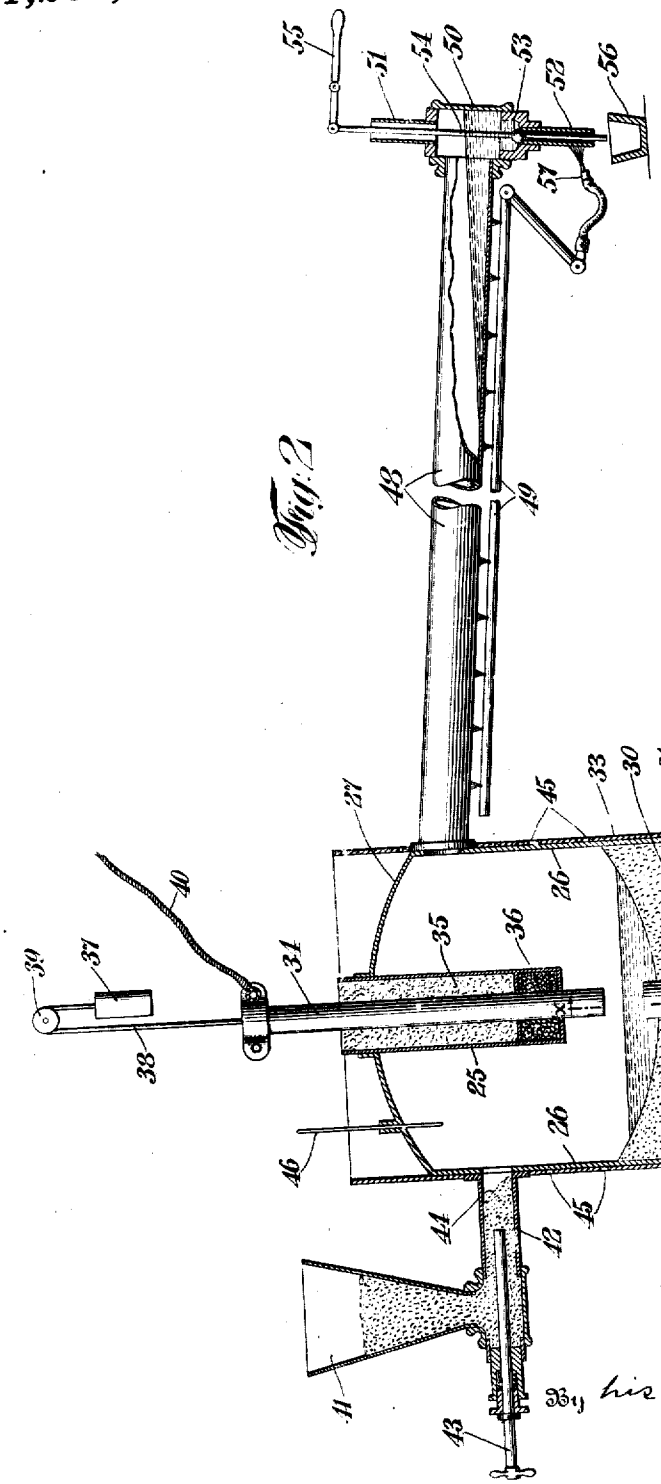
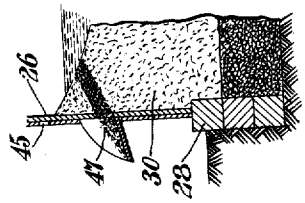


Fig. 3



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3 SHEETS—SHEET 3.

Fig. 4

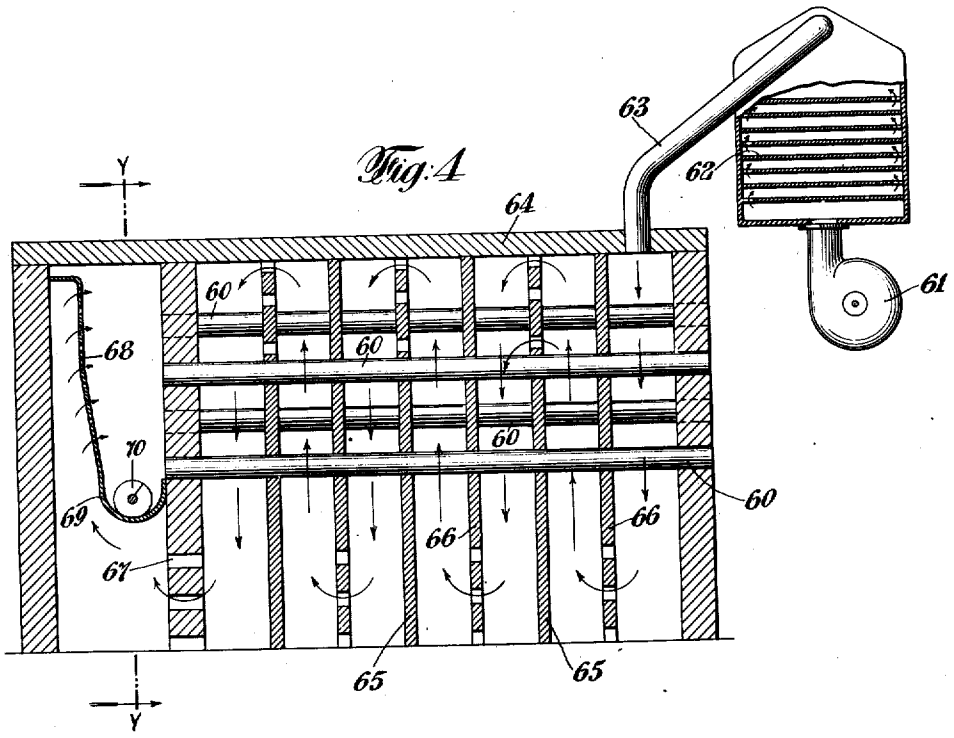
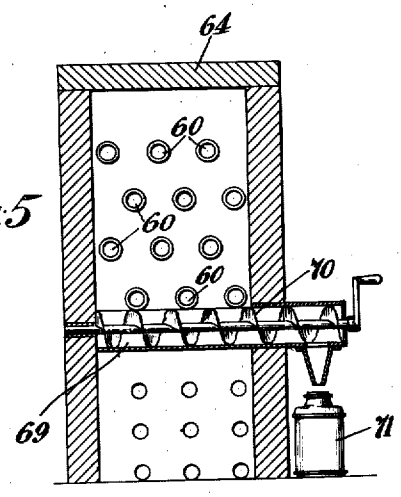


Fig. 5



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UNITED STATES PATENT OFFICE.

WALTER L. MORRISON, OF CANONSBURG, PENNSYLVANIA, ASSIGNOR TO ELECTRIC REDUCTION COMPANY, A CORPORATION OF DELAWARE.

PROCESS FOR PRODUCING ALKALI METALS OR THE LIKE.

1,265,360. Specification of Letters Patent. Patented May 7, 1918.

Application filed August 17, 1916. Serial No. 115,382.

To all whom it may concern:

Be it known that I, WALTER L. MORRISON, a citizen of the United States, residing at Canonsburg, county of Washington, State of Pennsylvania, have invented certain new and useful Improvements in Processes for Producing Alkali Metals or the like, of which the following is a specification.

The invention relates to an improved process for producing metals, and more particularly for the production of alkali metals, such as sodium and potassium. The main object of the invention is to provide an improved process for the obtaining of such metals in a simple and efficient manner, rapidly and without expensive apparatus. More specific objects, features and advantages will more clearly appear from the detailed description given below, taken in connection with the accompanying drawings, which form a part of this specification.

In the drawings Figure 1 illustrates an apparatus, partly in section, in which the preliminary treatment of the material may be carried out. Fig. 2 illustrates an apparatus, partly in section, in which the material obtained from the apparatus of Fig. 1 is heated and the vapor of the metal produced and condensed. Fig. 3 is a framentary section of a part of the apparatus shown in Fig. 2, taken on the line X—X of Fig. 2. Fig. 4 is a view, mostly in section, of an apparatus, by means of which the metal obtained from the apparatus in Fig. 2 may be oxidized to peroxid. Fig. 5 is a section taken on the line Y—Y of Fig. 4.

While the improvements are adapted to the production of various metals and peroxids thereof, yet they are particularly applicable to the production of alkali metals, and in order that the invention and its application may be clearly understood, I will describe them as applied to the production of metallic sodium and peroxid of sodium.

While any suitable compound of sodium may be used as a starting material, I prefer to use caustic soda or sodium hydrate, because of its cheapness and the ease with which it lends itself to the process. The sodium hydrate I mix with a suitable dehydrating agent. For this purpose I preferably use silicon in the form of ferrosilicon (90% silicon). Both of these materials are first preferably finely ground and then mixed in the molecular proportion of

$$4NaOH+1Si$$

This mixed mass may be then caused to react by touching the same with a red hot iron. The vigorous reaction which results progresses throughout the mass, liberating large quantities of hydrogen and rendering the mass red hot. The hydrogen may be collected and used for any desired purpose. The reaction which occurs is believed to be as follows:

$$4NaOH+1Si=Na_2OSiO_2+4H+Na_2O.$$

Thus a basic sodium silicate is formed, together with some sodium oxid, which is more or less combined with the basic sodium silicate. The mass, on cooling, remains as a granular sandy residue, which is ground to a fine powder, and is then ready for the second step or treatment of the process.

This first treatment, as above explained, may be carried out in any suitable receptacle, such as a large cast iron pan, but if it is desired to have the process continuous, and collect the hydogen produced, an apparatus such as shown in Fig. 1 may be used.

Referring to Fig. 1, 1 represents a cast iron chamber provided with a water jacket 2, for keeping the same cool. The chamber 1 is provided with a top 3, having a cover 4 which may be removed for the introduction of the material. The upper part of the chamber is provided with a water-jacketed partition 5, having an opening 6 adapted to be closed by a valve 7, operated by a lever 8. Below the partition 5 is another water-jacketed partition 9, having an opening therethrough adapted to be closed by a valve 10, operated by a lever 11. Below the partition 9 are water-jacketed protecting baffles 12.

Leading from the top of the apparatus is a pipe 13, which is connected to a vacuum pump. In the bottom of the apparatus there is arranged a cut-flight conveyer 14 of heavy construction, extending laterally through a cooling tunnel 15, having connected at its other end a pipe 16, provided with a gate valve 17, leading to an air-tight container 18, provided with an outlet 19 at its lower end, normally closed by a valve 20. A by-

pass 21 for gases is provided around the valve 17, which by-pass may be closed by valve 22. Leading laterally from the chamber 1 is a water-cooled conducting pipe 23, which extends into the bottom of a suitable gas tank 24, for the storage of the hydrogen gas produced by the process.

The caustic soda and ferro-silicon being introduced through the opening covered by the cover 4, fall upon the partition 5. Upon replacing the cover 4, the air is exhausted from the space above the partition 5, then the valve 7 is operated to allow the material to fall upon the partition 9 and the air again exhausted. The valve 7 being closed, valve 10 is then opened and the material falls upon the baffles 12, and from them drops to the bottom of the chamber 1.

The reaction may be initiated by heating this material by passing current through an electrical resistance wire extending into the bottom of the chamber 1, and after the reaction is thus once initiated, then, as further material is introduced into the chamber, as above explained, it falls upon the hot material at the bottom and the reaction is thus substantially continuous. The cut-flight conveyer 14, which is of low pitch, more or less cuts up the lumps of the material and at the same time feeds it to the pipe 16, so that it drops into the container 18. When it is desired to remove the material from the container 18, the valve 17 is closed, and the valve 20 opened, and the material allowed to drop into any suitable car or other receptacle 25, the valve 22 being left open sufficiently to permit enough hydrogen gas to pass into the chamber 18, to replace the material removed therefrom. The hydrogen gas produced by the reaction passes from the cooling pipes 23 and is then stored in the gas tank 24, to be used as desired.

The basic silicate obtained in the first step of the process is then mixed with a suitable slag-forming element, such as lime, magnesia or other material of calcareous nature, and a suitable reducing agent, such as silicon (ferro-silicon may be used), in about the following molecular proportions:

$$3CaO+Si+(Na_2O)_2SiO_2= 4Na+(CaO)_3(SiO_2)_2$$

All of the materials are finely ground. This mixture is then fed into an electric furnace and the mass heated. When a temperature of about 2000° F. is reached, the mass reacts and it is believed forms a calcium silicate, containing a small proportion of sodium silicate, but most of the sodium is liberated in the form of metal vapor, which distils out of the furnace and is then condensed in a suitable condenser. The temperature of the condenser may be so regulated as to hold the metallic sodium in the form of a liquid until a suitable quantity thereof is condensed, and it may then be tapped off into molds, or otherwise conveniently removed. The condenser is preferably therefore kept at approximately the temperature of boiling water, to keep the sodium in a molten condition. A suitable apparatus for carrying out this second step is illustrated in Figs. 2 and 3.

The electric furnace comprises a wrought iron cylindrical container 26, of about one-half inch thickness, having an iron top member 27, and with its lower edge resting upon cylindrical brick work 28. Within the cylindrical brick work 28 is tamped a bed of coke 29, and above this a bed of lime 30, which forms the bottom of the furnace. Extending up through the bed of lime and normally protected by the superposed slag is one electrode 31 of the furnace, which at its bottom is electrically connected to an iron bar 32, which in turn is suitably connected to a source of alternating electric current. Wrapped around the iron cylinder 26 are about eight layers of asbestos paper 33. It has been found that electrodes of a character adapted to conduct current economically and withstand the necessary temperature to apply electrical energy to the mass, such for instance as graphite electrodes, are rapidly attacked by the cooling metal vapors at points removed from the hot ends of the electrodes if the metal vapors are allowed to come in contact therewith and particularly wherever the metal vapor is permitted to condense in or on the electrode. The other electrode is shown at 34, and in order that this may be protected from the action of the metallic vapor, it is surrounded by a thick layer of powdered lime 35, and at its bottom portion by a thick layer 36 of powdered lime containing about 10% of finely powdered ferro-silicon, the lime and ferro-silicon being held in place by an iron shell. The carbon or graphite electrode 34 may be adjusted by means of a weight 37, connected to the electrode by means of a rope 38 passing over a pulley 39. The electrode is suitably connected to the source of electrical energy, by means of a flexible conductor 40. The cylindrical member 26 is preferably about thirty inches high and thirty inches in diameter.

The mixture of basic silicate, lime and reducing agent is fed into a hopper 41, leading to inlet pipe 42, into which extends a plunger or ram 43, which may be operated back and forth by hand, to gradually feed the material 44 from the pipe 42 into the electric furnace. One method of judging whether the temperature in the furnace is at the desired point, is to observe the color of the wrought iron shell 26. For this purpose holes 45 are cut through the asbestos paper 33, so that the wrought iron may be seen. When the mixture is heated in the electric furnace to about 2000° F., the vapor

of metallic sodium is produced, leaving calcium silicate as a slag and this slag may be tapped off from time to time, through the slag opening 47 (see Fig. 3), which slag opening may be filled up with lime or powdered slag when it is not in use. The metallic vapors are conducted to a condenser 48, consisting of a six inch wrought iron pipe connected directly with the cylinder 26, and being inclined downwardly about one inch to the foot, and being about twelve feet long. In order to keep this pipe at the proper temperature, to maintain the condensed sodium in liquid form, a gas pipe 49 is provided therebeneath, with gas-jets impinging gas flames upon the pipe 48 to keep it at the desired temperature. Connected at the lower end of the pipe 48 is a T-head 50, having a two inch pipe 51 leading from the top thereof, and a three-quarter inch pipe 52 leading from the bottom thereof, the top of the pipe 52 being adapted to be closed by a ball-valve 53 carried upon a stem 54, operated by a lever 55. Upon raising the ball-valve 53, the metallic sodium in liquid form will run into the cast iron molds 56 placed beneath the pipe 52 for collecting the same. Should the metallic sodium tend to freeze in the pipe 52, or above the ball-valve 53, a gas jet 57, flexibly connected, may be directed against these parts in order to melt the metallic sodium and cause it to flow freely down to the molds 56. In view of the fact that there may be some little caustic soda which has not been transformed into basic silicate in the first step, small amounts of hydrogen gas may be given off from the electric furnace, which gas will escape through the pipe 51 and may be burned at the end thereof. The lower end of the pipe 48 should be kept at about 100° C. while the upper end may be red hot from the condensed sodium. The molds 56 may be wiped inside with a little oil and graphite, to permit of the easy removal of metallic sodium therefrom.

If it is desired to convert metallic sodium into peroxid, the apparatus shown in Figs. 4 and 5 may be used. Metallic sodium may be fed by hand into the pipes 60, at the right hand end, while hot air is passed through the pipes from the left hand end, causing the metallic sodium to burn to sodium peroxid. The air is first forced by means of a suitable fan or pump 61 over trays 62 in a suitable container, which trays contain lime or caustic soda in order to dry and purify the air. On leaving the trays 62, the air is forced through a pipe 63 into the right hand end of a chamber 64, surrounding the pipes 60. This chamber is provided with suitable baffles 65 and 66, which baffles have openings alternately at the top and bottom, so as to cause the air from the pipe 63 to pass back and forth transversely across the pipes 60, and thereby become heated. Upon reaching the left hand end of the apparatus, the warm air passes through apertures 67 and then through a perforated sheet 68 and into the left hand end of the pipes 60. The material is gradually fed through the pipes 60 from right to left, and shown in Fig. 4, by hand, by means of a two-tined rake and drops from the left hand end of the pipes 60 as peroxid, into a trough 69, from which it may be removed by a screw conveyer 70 into suitable cans or containers 71. The rate at which the air is introduced or forced through the apparatus by the fan is so regulated as to maintain the temperature of the air as it passes through the perforated plate 68 at about 150° C., while the temperature of the nitrogen and small quantities of oxygen leaving the pipe 60 at the right hand end should be about 250° C.

It will be seen from the above that the improved process in its preferred form contemplates taking any suitable compound of an alkali metal or the like, and heating it with any suitable reducing agent to a temperature sufficient to produce the vapor of the metal and then condensing the vapor. By so choosing the compound and reducing agent that no gases are produced other than the metallic vapor, very serious difficulties are avoided. The gases would not only dilute the vapor, but it would be very difficult to separate the gases from the metal vapor and other serious difficulties, as a manufacturing proposition, would result. Hence it is preferred to treat the starting material first with a de-hydrating agent to remove the hydrogen from the material. By this preliminary treatment it is possible to make metallic alkali metal from the hydrates thereof, in a very simple and efficient manner. If the starting material contains no hydrogen or other gas forming elements, which would be disadvantageous in the second step, it is only necessary to choose a suitable reducing agent which will not cause such gases to be produced in the electric furnace, and for this purpose it is preferred to use a non-carboniferous reducing agent, such as silicon, as it then will not produce the oxids of carbon.

It will be understood that many changes and modifications may be made without departing from the spirit and scope of the invention in its broader aspects. Hence it is desired to cover all processes coming within the language of the appended claims.

What is claimed and desired to be secured by Letters Patent is:

1. The process of producing alkali metals which consists in heating a compound of an alkali metal with a reducing agent to produce alkali metal vapor, the alkali metal compound and silicious reducing agent being so chosen that substantially no gases, as

distinguished from metal vapor are produced by the reaction, and then condensing the alkali metal vapor.

2. The process of producing alkali metals which consists in heating a basic silicate of the alkali metal with a reducing agent to produce alkali metal vapor, the reducing agent being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the alkali metal vapor.

3. The process of producing alkali metals which consists in heating a compound of an alkali metal with a silicon non-carboniferous reducing agent, the alkali metal compound being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the alkali metal vapor.

4. The process of producing alkali metal which consists in heating a basic silicate thereof with a non-carboniferous reducing agent, to produce alkali metal vapor and then condensing the vapor.

5. The process of producing sodium which consists in heating a basic sodium silicate with a non-carboniferous reducing agent, and calcareous material, to produce metallic sodium vapor and then condensing the sodium vapor.

6. The process of producing metals from compounds thereof, which consists in heating in an electric furnace a basic silicate thereof with a suitable reducing agent sufficiently to vaporize the metal therefrom, the compound and reducing agent being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the metal vapor.

7. The process of producing alkali metals from compounds thereof, which consists in carrying out two steps; in the first step, treating the alkali metal compound to alter it so that those elements are removed therefrom which would produce gases in the second step, and in the second step, heating the so prepared material with a reducing agent to vaporize the metal and condensing the metal.

8. The process of producing alkali metals from compounds thereof, which consists in dehydrating the compound, then heating the same in the presence of a reducing agent to produce alkali metal vapor and condensing the vapor.

9. The process of producing alkali metals from hydrated compounds thereof, which consists in dehydrating the compound, then heating it in the presence of a reducing agent to produce alkali metal vapor, the reducing agent being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the alkali metal vapor.

10. The process of producing alkali metals from hydrated compounds thereof, which consists in dehydrating the compound, then heating it in the presence of a non-carboniferous reducing agent, to produce alkali metal vapor and condensing the vapor.

11. The process of producing alkali metals from hydrated compounds thereof, which consists in dehydrating the compound, then heating it in the presence of a non-carboniferous reducing agent, and lime, to produce alkali metal vapor and condensing the vapor.

12. The process of producing alkali metals from alkali metal hydrates which consists in treating the hydrate with silicon to produce a basic silicate of the alkali metal and then heating the basic silicate with a non-carboniferous reducing agent, to produce alkali metal vapor and condensing the vapor.

13. The process of producing alkali metals from alkali metal hydrates which consists in treating the hydrate with silicon to produce a basic silicate of the alkali metal and then heating the basic silicate with a reagent to produce alkali metal vapor and condensing the vapor.

14. The process of producing alkali metals from alkali metal hydrates which consists in treating the hydrate with silicon to produce a basic silicate of the alkali metal and then heating the basic silicate with a reducing agent to produce alkali metal vapor, the reducing agent being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the alkali metal vapor.

15. The improved process which consists in heating a compound of an alkali metal with a reducing agent to produce alkali metal vapor, the alkali metal compound and reducing agent being so chosen that substantially no gases, as distinguished from metal vapor, are produced by the reaction, and then condensing the alkali metal vapor and burning the metal to produce the peroxid thereof.

16. The improved process which consists in heating a basic silicate of an alkali metal with a non-carboniferous reducing agent, to produce alkali metal vapor, and burning the alkali metal to form peroxid thereof.

17. The process of producing alkali metals from compounds thereof, which consists in treating the compound with a reagent to produce a basic silicate thereof, and then heating the silicate with a non-carboniferous reducing agent to produce alkali metal vapor.

18. The process of producing alkali metals from compounds thereof, which consists in treating the compound with a reagent to produce a basic silicate thereof, and then heating the silicate with a non-carboniferous reducing agent and lime to produce alkali metal vapor and condensing the metal vapor.

19. The process of producing metal and hydrogen from compounds thereof, which consists in treating the compound with a reagent to remove the hydrogen in the form of a substantially pure gas and then treating the residue with a reducing agent to vaporize the metal and then condensing the metal.

20. The process of producing alkali metals which consists in heating a suitable compound thereof by means of electric current applied by electrodes adjacent the compound, to produce the metal in vapor form and protecting the electrodes from the metal vapor in order to permit of the vapor being condensed and recovered.

In testimony whereof, I have signed my name to this specification.

WALTER L. MORRISON.

Corrections In Letters Patent No. 1,265,360.

It is hereby certified that in Letters Patent No. 1,265,360, granted May 7, 1918, upon the application of Walter L. Morrison, of Canonsburg, Pennsylvania, for an improvement in "Processes for Producing Alkali Metals or the Like," errors appear in the printed specification requiring correction as follows: Page 1, line 29, for the word "framentary" read *fragmentary;* page, 3, line 127, claim 1, before the word "reducing" insert the word *silicious;* and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 28th day of May, A. D., 1918.

[SEAL.]

R. F. WHITEHEAD,

Acting Commissioner of Patents.

Cl. 204—63.