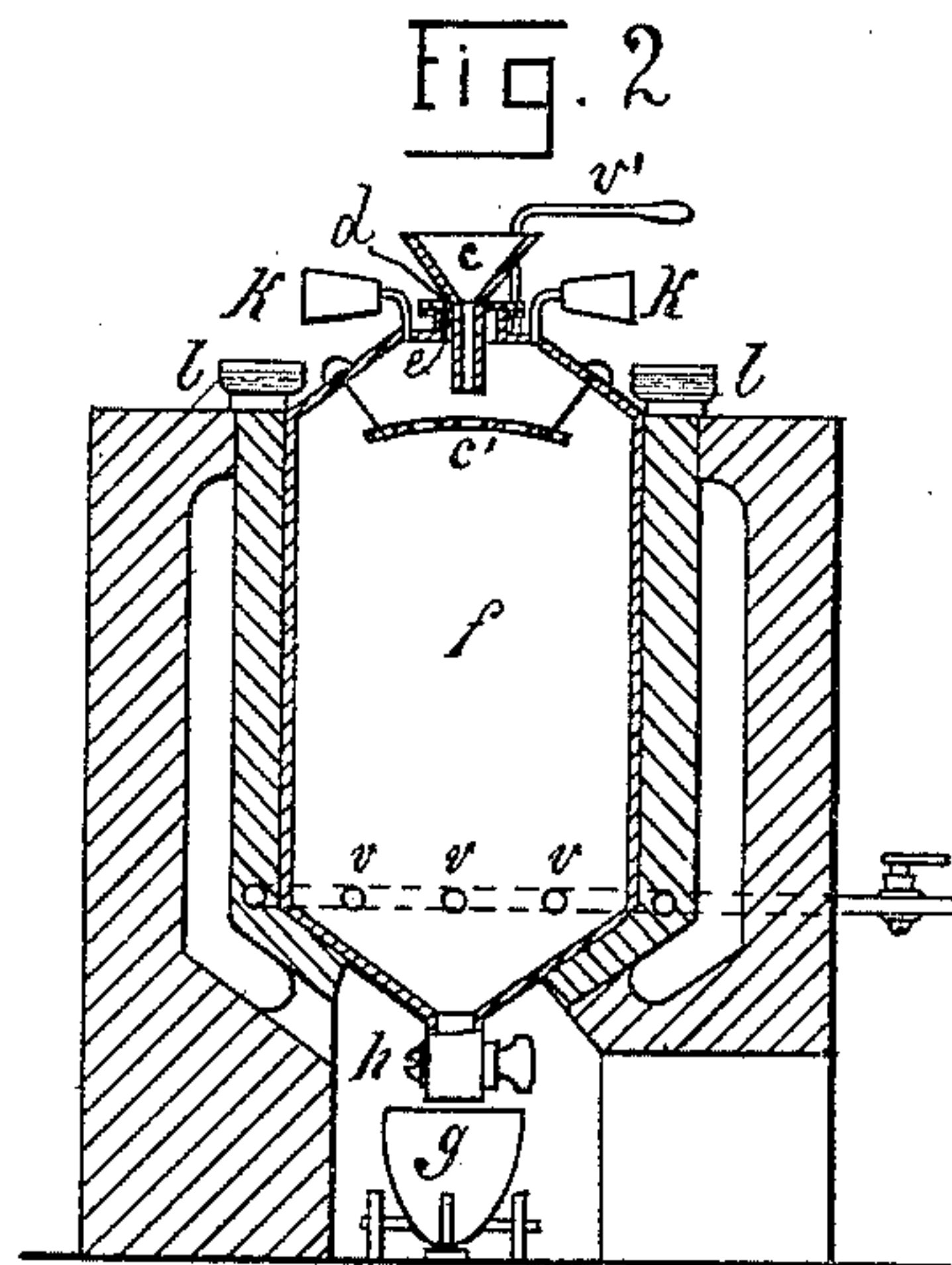
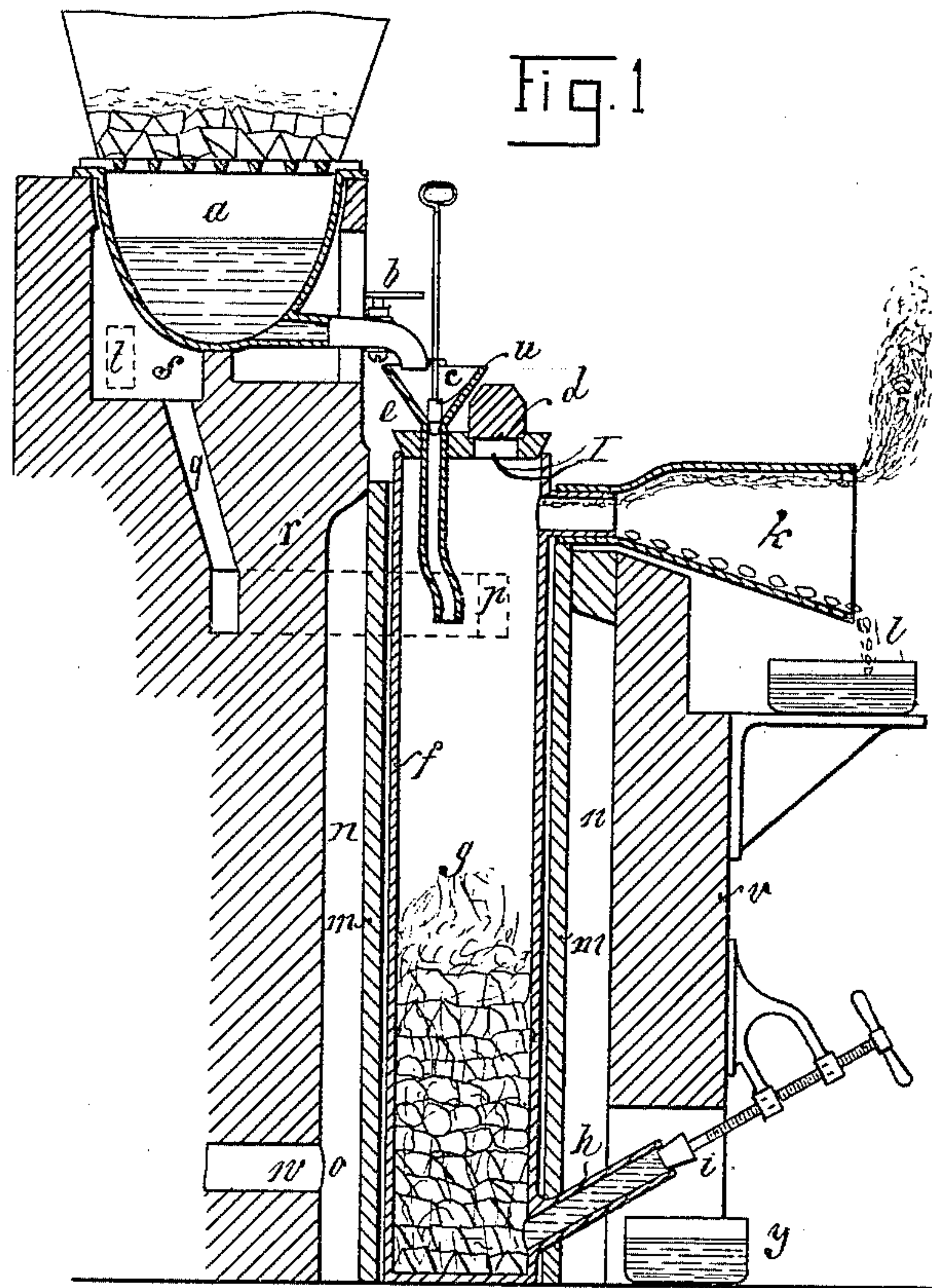


(No Model.)

C. NETTO.
PROCESS OF MAKING SODIUM OR POTASSIUM.
No. 460,985. Patented Oct. 13, 1891.



Witnesses.
A. B. Blackwood
H. Smith

Inventor
Curt Netto
By Comstock Bros
Atty's

UNITED STATES PATENT OFFICE.

CURT NETTO, OF DRESDEN, SAXONY, GERMANY.

PROCESS OF MAKING SODIUM OR POTASSIUM.

SPECIFICATION forming part of Letters Patent No. 460,985, dated October 13, 1891.

Application filed February 14, 1888. Serial No. 263,949. (No specimens.) Patented in England October 26, 1887, No. 14,602; in Germany November 5, 1887, No. 45,105; in France December 1, 1887, No. 187,349; in Belgium December 1, 1887, No. 79,759, and in Austria-Hungary August 16, 1889, No. 7,693 and No. 40,262.

To all whom it may concern:

Be it known that I, CURT NETTO, engineer, a subject of the King of Saxony, residing at Dresden, Saxony, German Empire, have invented certain new and useful Improvements in the Manufacture of Sodium or Potassium and in Apparatus Therefor, (for which I have obtained the following patents: in Austria-Hungary, No. 7,693 and No. 40,262, dated August 16, 1889; in England, No. 14,602, dated October 26, 1887; in Germany, No. 45,105, dated November 5, 1887; in Belgium, No. 79,759, dated December 1, 1887, and in France, No. 187,349, dated December 1, 1887;) and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same, reference being had to the accompanying drawings, and to letters of reference marked thereon, which form a part of this specification.

The present invention has relation to a continuous process for the manufacture of alkali metals, as potassium, sodium, &c., and the production at the same time of alkaline carbonates from the hydroxides of the respective metals by reduction with reducing-agents, like coal or other carboniferous materials.

For the manufacture of sodium (sodium will be chosen here as an example) by the reduction of caustic soda by means of coal the result of the reduction when complete finds its expression in the following equation: $\text{NaHO} + \text{C} = \text{Na} + \text{H} + \text{CO}$. Now in the manufacture of the sodium from caustic soda it will be observed that at the beginning the separation of the sodium metal is effected at a comparatively low temperature; but that very soon, in order to maintain the separation, the employment of higher temperature becomes necessary, much to the detriment of the vessels employed in the reduction and greatly in increase of the consumption of fuel. The course of this process shows, therefore, two different phases. In the first, wherein the reduction is effected at a comparatively low temperature, about red heat, the formation of carbonate of soda, metallic sodium, carbonic oxide, and hydrogen can be expressed by the equation: $4(\text{NaHO}) + 2\text{C} = \text{Na}_2\text{CO}_3 +$

$2\text{Na} + 4\text{H} + \text{CO}$. This first phase being completed, the remaining carbon will not have to act upon the carbonate of soda formed in the first phase. This requires a considerably higher temperature than is necessary for the first phase. The second phase, comprising the process of reduction of the carbonate of soda formed in the first phase, may find its expression in the following equation: $\text{Na}_2\text{CO}_3 + 2\text{C} = 2\text{Na} + 3\text{CO}$. This indicates that the temperature necessary for the reduction must be more and more elevated, accordingly as the formation of carbonate of soda increases. This inconvenience occurs also in the known process which consists in throwing the caustic alkali to be reduced upon the previously-heated coal. At first the reduction proceeds at a comparatively low temperature; but subsequently the temperature must be raised more and more until finally an interruption of the process becomes necessary in order to allow the molten mass or the scoria to be scraped or removed from the reducing-vessel. It is even necessary sometimes to provide a new reducing-vessel, as the one in use can be quickly destroyed by the molten mass and the higher temperature required after the formation of carbonate of soda.

It is the object of the present invention to obviate the inconvenience above stated, and such invention contemplates the manufacture of the alkali metals in the first phase of the process of reduction in order to have to deal with comparatively low temperatures.

In conformity with the above-mentioned observations and considerations the alkaline carbonate first formed by the reduction of caustic alkali by means of carboniferous materials will naturally mix with the caustic alkali, so that the temperature required for the reduction necessarily becomes higher and higher. To avoid this the process forming the object of the present invention is so carried into effect that, in the first place, the mixture of the alkaline carbonate formed during the first part of the process with the caustic alkali is to a great extent avoided, and, in the second place, the carbonate produced in the first stage of the process is led away during the process of reduction continuously, or from time to time, as may be necessary, while

the caustic alkali is gradually and, if possible, continuously added to the sufficiently-heated reducing material—*i. e.*, to the heated coal—the supply of coal being renewed accordingly as it becomes exhausted in the retort.

Care should be taken that the resultant volatilized sodium (if sodium be the metal sought) is always immediately led off, and that the replacing of the used reducing material can be effected without interruption of the operation or the process. My process for obtaining alkali metals differs from the present known process, therefore, by the regular discharge of the formed carbonate and by the continual charge of the sufficiently-heated reducing material and of the caustic alkali mass to be reduced, so that the reducing action will take effect only upon the caustic alkali, and that the temperature of the reduction consequently need be only a low one.

An apparatus represented as an example on the accompanying sheet of drawings is appropriate to be employed for the working of the present process, where the caustic soda in liquid state and in a continual jet shall flow or drip upon the glowing coal present in excess.

In the drawings, Figure 1 is a vertical sectional view, and Fig. 2 a similar view of a modification.

a represents the reservoir used for melting the caustic soda, the outlet-pipe of which is provided with a regulating-faucet *b*.

c is the charging-funnel for the molten caustic soda, which leads through the lid *e*, the latter being provided with a charge-hole *I* for the coal into the retort *f*, made of proper material—*i. e.*, cast-steel, wrought or cast iron—and wherein the reduction should take place under the influence of the coal *g*. The tap-pipe *h*, arranged near the bottom of the retort, is properly provided with a valve *i*. In an opening at the upper part of the retort a receiver *k*, somewhat narrow, is properly arranged for condensation of the sodium. Under the outlet of this condenser is placed a reservoir *l*. The retort *f* is covered with a jacket *m* of clay. The gases used for heating are led through the flue *w* and opening *o* into flue *n*, where they circulate around the retort. Then the gases pass through the opening *p*, flues *r* and *q* into the heating-space *s*, where they heat the vat *a*, and now they can be led off through opening *t* into the chimney. The charging-funnel *c* can be provided with a conical valve *u*. Below the tap-pipe *h* is placed a receiving-vessel *y* for the molten mass of soda formed during the reduction.

By means of such apparatus the present process is executed in the following manner: The retort is heated to about cherry-red, then it is charged by either taking off the lid *e* or through the charge-hole *I* with as much coal, preferably charcoal, as will fill about one-third of its capacity. When the coal is glowing, (a few minutes are sufficient for the pur-

pose,) the molten caustic soda is allowed to run from the vat *a* through the funnel *c* upon the glowing coal. The flow can be regulated by the faucet *b*. Immediately the sodium flame becomes visible at *k*, and after a very short time the sodium flows off from *k* into the oil-reservoir *l*. In the beginning it is necessary to close the valve *i* in order to keep off the atmospheric air; but in a short time, about half an hour, enough liquid mass, the greater of which is carbonate of soda, will have gathered at the bottom of the retort, so as to form at *h* a hydraulic closure, and now the valve *i* can be opened, whereupon any excess of molten mass can freely flow off through the tap-pipe *h*. When after some hours the stock of coal in the retort is nearly exhausted, the flow of caustic soda is cut off by closing the faucet *b*, and after waiting some minutes, until the sodium flame at *k* has nearly disappeared, fresh coal is charged in through *I*, whereupon the operation recommences after having closed the opening *I*, which can be done, for instance, by simply placing upon it an iron cylindrical plug. It is obvious that the introduction of coal may be effected in any way—*i. e.*, as in charging gas-generators—in order to completely avoid opening the retort for charging coal. It is not necessary to fasten the lid *e* by screws or wedges. It is sufficient to simply lay it in place. Any other carboniferous material may just as well be employed for the decomposition; but charcoal offers a special advantage on account of its containing a low degree of silicic acid and a comparatively high degree of alkali in its ashes. On account of the absence of silicic acid stubbornness of the liquid mass is avoided, and by reason of the comparatively high degree of alkali in its contents the losses of alkali become comparatively small, notwithstanding the unavoidable sodium flame. The mass can be separated from particles of coal contained in it by lixiviation and the solution can be worked to carbonate of soda or regenerated into caustic soda.

The process can be executed also in an apparatus where the heating of the retort is not effected from the outside (when similar to the process of manufacturing water-gas an intermittent periodical working is adopted) by alternately causing the coal contained in the apparatus to glow under access of air or other gases and introducing the soda, then again leading air or burning gases into the apparatus to heat the coal, then again bringing in soda, &c. For this purpose the apparatus represented by Fig. 2 is appropriate. The large vertical cylinder *f*, made of boiler-sheets and matted in with brick-work, is provided on its head-plate with a number of reservoirs *K*, wherefrom the condensed alkali metal can flow into the oil-reservoir *l*. It is further provided with an opening *I*, which can be closed by a lid *e*, to throw in the coal and with a charging-funnel *c* for the molten soda. To the conical bottom of the cylinder is attached a tap-pipe

5 *h*, having a means to close it, by which the
 formed products can flow into a receptacle
 placed below, which is best provided with
 wheels, while near the bottom tuyeres *v* are
 arranged, through which air or burning gases
 may be introduced for the purpose of heating
 the coal. The upper head-plate may also be
 provided with a siphon-pipe *v'*. The funnel-
 pipe of the charging-funnel *c* is best provided
 10 with a perforated curved or conical divider
 to divide the molten mass of soda. The ap-
 paratus is filled with glowing charcoal, and
 then air, or a mixture of air and inflammable
 gas, is blown in till the necessary temperature
 15 is obtained. Then the flow of gases is cut off
 and the caustic soda is admitted as long as
 any sodium separates in the apparatus, where-
 upon the gases in it are drawn off, and then
 the necessary temperature is re-established by
 20 the introduction of air or burning gases. If
 it be desired to accelerate the process in this
 apparatus by heating from outside, the brick-
 work surrounding the cylinder can be pro-
 vided with fire or gas flues. The inner heat
 25 can be produced either by introducing air to
 the glowing coal, thus producing combustion,
 or by introducing inflammable or burning
 gases or mixtures of gases. As soon as the
 coal is consumed, fresh coal is filled in, which
 30 can be effected by simply throwing it in, or in
 any other way—*i. e.*, in a manner similar to
 that employed in charging gas-generators.

In this process thus illustrated by different
 examples it is of course not necessary to in-
 35 troduce the soda in liquid state, though it
 seems to be most convenient and advanta-
 geous. It can as well in special cases be intro-
 duced in solid blocks. Moreover, instead of
 pure caustic soda impure sorts may be em-
 40 ployed—*i. e.*, what is known as "bottoms."

The advantages obtained by my present process are in short the following:

45 First. The process is carried on, as ex-
 plained above, at an equal comparatively-
 low temperature—*i. e.*, cherry-red heat. The
 retorts, therefore, are not attacked, and hence
 wear a long time, so that even cast-iron can
 be employed as the material in building them.

50 Second. As the retorts remain in the kiln
 and are not withdrawn, as in the case with
 the crucibles in other processes, the dimen-
 sions of the same can be considerable and
 they can be covered with preserving-coating
 of clay.

55 Third. By the employment of vertical re-
 torts the disadvantages of horizontal retorts
 consisting in a slight sagging of the same,
 whereby the preserving clay coat falls off, are
 avoided.

60 Fourth. As the lid of the horizontal retorts
 has to be screwed on, and as the highly-heated
 gases formed in the retort cannot escape after
 the openings leading to the condensers *k* are
 obstructed, the danger of explosion becomes
 65 imminent and explosions have in fact occurred

on account of the lively development of hy-
 drogen in the beginning. This danger of ex-
 plosion is completely avoided in the present
 process, as the quantity of the soda introduced
 can at all times be regulated by the position 70
 of the faucet *h*, or completely shut off. More-
 over should the pressure in the retort by any
 reason—say, by negligence—become too high,
 the iron plug *d* at *I* operates like a safety-
 valve, and will be lifted by the excess of power, 75
 so that the gases can freely escape.

Fifth. The present process is very simple
 and convenient compared to the other known
 processes, wherein it is very disagreeable,
 even dangerous, to handle an obstructed re- 80
 ceiver when luted by the heat, for even when
 removed burning sodium vapors under press-
 ure rush upon the workman, and if he should
 not succeed in removing the obstruction in a
 few minutes, nothing can be done except 85
 abandon the retort and quench the fires. Agair,
 this disagreeable evacuation of the re-
 torts and the charging of the same, the fixing
 of the lids on the hot retorts during the oper-
 ation are not necessary with the present pro- 90
 cess, wherein by a simple closing off of the
 faucet in the alkali supply-pipe the action
 ceases by itself after one or two minutes.

The process constituting my invention ad-
 mits of the use of less valuable products con- 95
 taining soda—*i. e.*, the bottoms—the use of
 which has heretofore been economically im-
 practicable, as the charge had to be held con-
 centrated as much as possible.

Seventh. The present process admits of a 100
 continuous operation with all its advantages,
 consisting in easy work, sparing of the re-
 torts and the coatings, avoiding the cooling
 off, with the bad influence produced upon the
 neighboring retorts in the kiln while charg- 105
 ing or discharging one retort, sparing of the
 kiln, economizing fuel, &c.

Having described my invention, what I claim is—

The process for the production of potassium 110
 or sodium from their hydroxides in a contin-
 uous operation, which consists in bringing the
 caustic alkali into contact with the reducing
 carboniferous matter at such a low tempera-
 ture that only the caustic alkali is reduced to 115
 a metallic state, while the alkali carbonate
 simultaneously formed remains undecom-
 posed and is withdrawn out of the reach of
 the reducing carboniferous matter without
 interruption of the reducing process or the 120
 admission of air to the reducing-vessel, the
 caustic alkali being charged continuously into
 the reducing vessel or retort during the re-
 ducing operation, substantially as described.

In testimony whereof I affix my signature in 125
 presence of two witnesses.

CURT NETTO.

Witnesses:

HERMANN KUHFS,
 FRITZ MOELLENHOFF.