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Patented May 15, 1900.

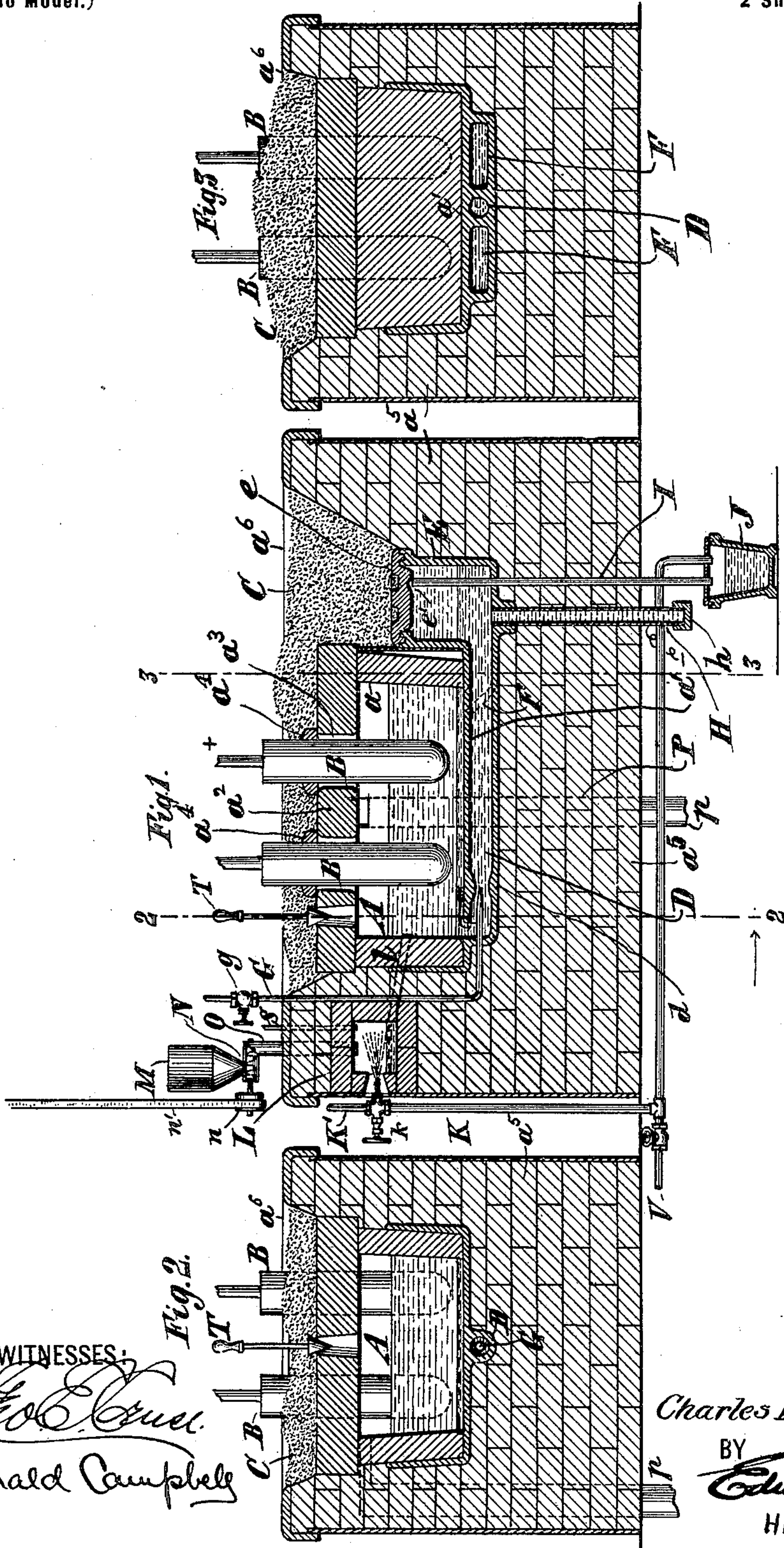
C. E. ACKER.

PROCESS OF MANUFACTURING CAUSTIC ALKALI AND HALOGEN GAS.

(Application filed Apr. 8, 1899.)

(No Model.)

2 Sheets—Sheet 1.



WITNESSES:

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*Edwin H. Brown.*  
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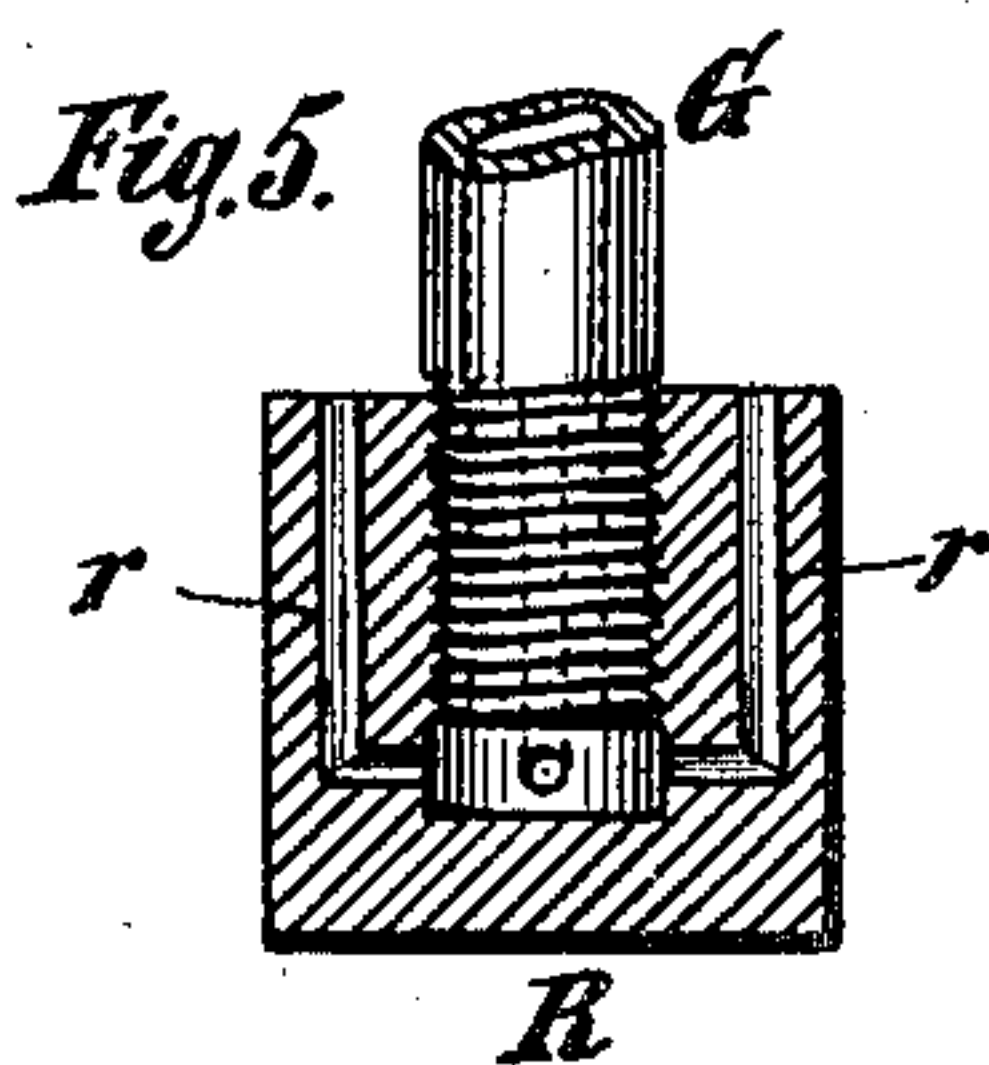
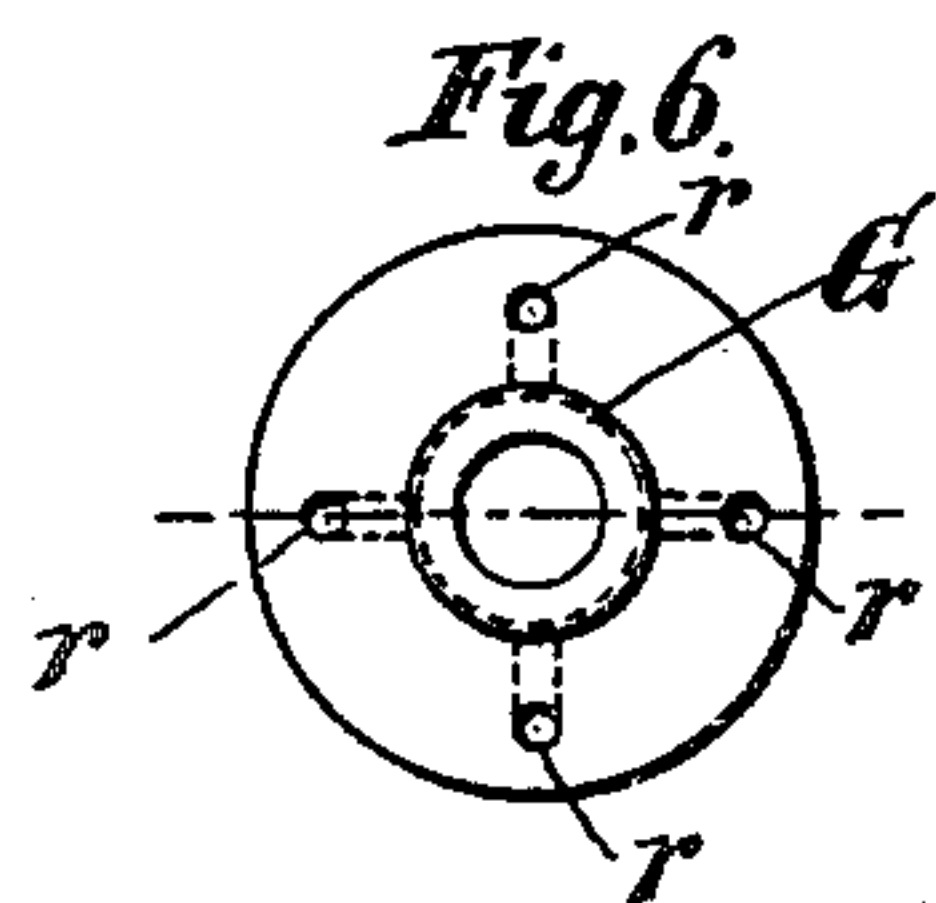
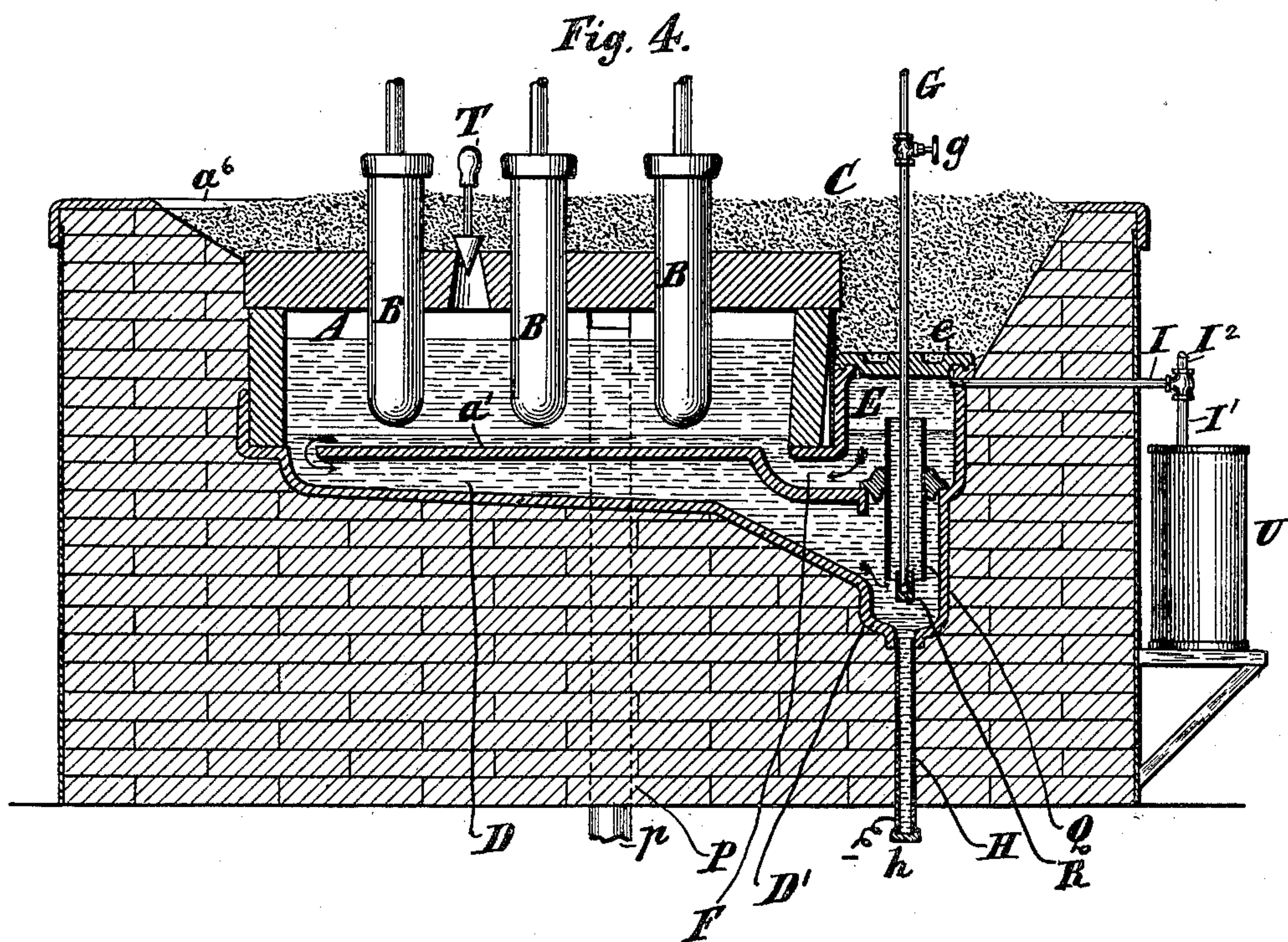
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# UNITED STATES PATENT OFFICE.

CHARLES ERNEST ACKER, OF EAST ORANGE, NEW JERSEY, ASSIGNOR TO  
THE ACKER PROCESS COMPANY, OF NIAGARA FALLS, NEW YORK.

PROCESS OF MANUFACTURING CAUSTIC ALKALI AND HALOGEN GAS.

SPECIFICATION forming part of Letters Patent No. 649,565, dated May 15, 1900.

Application filed April 8, 1899. Serial No. 712,207. (No specimens.)

*To all whom it may concern:*

Be it known that I, CHARLES ERNEST ACKER, a citizen of the United States, residing at East Orange, in the county of Essex and State of New Jersey, have invented a new and useful Improvement in Processes for the Manufacture of Caustic Alkali and Halogen Gas, of which the following is a specification.

The object of my invention is to economize in the production of practically anhydrous caustic alkali and halogen gas. My invention consists in a novel process for the purpose.

In the accompanying drawings, Figure 1 is a longitudinal vertical section of an apparatus for the manufacture of caustic alkali in accordance with my improvement. Fig. 2 is a transverse section of the same at the line 2 2, Fig. 1, certain parts being omitted. Fig. 3 is a transverse section of the same at the line 3 3, Fig. 1. Fig. 4 is a longitudinal vertical section illustrating a modification. Fig. 5 is a vertical section of certain parts upon an enlarged scale. Fig. 6 is a horizontal section of the same parts upon the same scale.

Similar letters of reference designate corresponding parts in all the figures.

Referring first to Figs. 1, 2, and 3, A designates an electrolytic furnace, which may be of any suitable form. As here shown, it has walls  $a$ , which may be made of basic material—as, for example, magnesia. These walls rest upon a hearth  $a'$ , which may be of iron or steel. The furnace is closed by a cover  $a^2$ , which may be made of fire-clay. This cover rests upon the walls  $a$ . In the cover are openings  $a^3$ , through which pass anodes B, preferably made of carbon, the openings  $a^3$  being, as here shown, considerably larger in diameter than the anodes. Preferably these openings will be closed around the anodes by auxiliary covers  $a^4$ , preferably made of two parts in the form of half-rings. Above the cover  $a^2$  and in the masonry  $a^5$  of the furnace a recess  $a^6$  is formed, and in this is a body of salt C. Below the hearth  $a'$  and communicating with the interior of the furnace A near one of the end walls of the latter is a conduit D. Preferably this will be cast integral with the hearth  $a'$ . It extends to the lower portion of a chamber E, and the latter

may be a portion of a single casting comprising the conduit and the hearth. The lower portion of the chamber E communicates by means of conduits F with that end of the interior of the furnace A which is opposite the end whence extends the conduit D. It will be seen by reference to Fig. 3 that the conduits F are arranged one on each side of the conduit D. A removable cover  $e$  closes the top of the chamber E, and the recess  $a^6$  extends down to such cover. Preferably the cover and chamber will have a double-lapped joint made by providing the cover on its under side with an annular rib that extends into an annular groove formed in the upper end of the chamber. A pipe H leads from the bottom of the chamber E and is provided with any suitable device for controlling the flow of its contents from it. As here shown, a removable cap  $h$  is employed for this purpose. This pipe may form the negative terminal of the furnace.

G designates a steam-pipe provided with a controlling-cock  $g$  and extending into the conduit D near that end of said conduit which is in communication with the interior of the furnace A. Preferably the conduit will have a contraction  $d$  near where the steam-pipe terminates, this construction being advantageous in order to produce an action analogous to that of an injector.

In the under side of the cover  $e$  of the chamber E is a recess  $e'$ . From the recess  $e'$  a pipe I, which may be made of iron or steel, extends downwardly to the outside of the furnace. As here shown, it extends directly down through the masonry. It communicates with a vessel J. From the top of the vessel J a pipe K leads to a burner, with which also communicates an air-pipe K' somewhat after the manner of an ordinary oxyhydrogen blow-pipe; but I utilize an air-blast to create a small suction for inducing a flow of hydrogen to the burner. A cock  $k$  controls the burner. This burner is comprised in a small auxiliary melting-furnace L, formed in the masonry of the furnace A and communicating with the furnace A by means of a passage  $l$ , preferably inclined, so that that end which communicates with the main furnace A will always be submerged in the contents of the latter. The



molten salt in this auxiliary furnace will normally be at the same level as the contents of the main furnace A. Whenever the level rises in the auxiliary furnace there will be a flow into the main furnace A. Owing to the fact that the level in the two furnaces will be normally the same, it is possible to ascertain the level in the main furnace by looking through the hole provided for the passage of the burner K into the auxiliary furnace, that hole being of sufficient size for the purpose. A pipe S, extending from the top of the auxiliary furnace, provides for carrying away gaseous products of combustion.

Salt to be treated in the furnace, if supplied through a hopper or chamber M and fed by a worm N into a pipe O, leading into the melting-furnace L, may be supplied to the electrolytic furnace in a molten condition at a uniform rate. A pulley  $n$  upon the shaft of the worm N derives motion from a belt  $n'$ , set in motion by a suitable motor. A plug T, fitting a hole in the cover  $a^2$  and provided with a handle extending above the body of salt C, may be manipulated for the purpose of permitting some of the body of salt C to flow into the main furnace A. Thus an independent feed will be possible. From the upper part of the interior of the furnace A a channel P extends through the masonry and communicates with a pipe  $p$ . It is intended that the body of salt C shall be maintained constantly in sufficient quantity in the recess  $a^6$  to form a seal for the openings to the covers  $a^2$  and  $e$ .

Lead in a molten state may be introduced through one of the anode-openings  $a^3$  in quantity sufficient to fill the conduit D and cover the hearth  $a'$ , an anode and its auxiliary cover  $a^4$  being for this purpose temporarily removed. Immediately afterward the salt—*e. g.*, sodium chlorid—may be introduced in a molten state in the same manner as the lead. Then the anode, with its auxiliary cover, will be replaced and the full current turned on. After the operation shall have been fully started salt is supplied by means of the hopper M, worm N, pipe O, and auxiliary furnace L, entering the main furnace A in a molten state. An alloy of sodium and lead will be formed. Steam under pressure escaping from the pipe G will produce a rapid circulation through the conduit D to the chamber E. It will be decomposed in transit within the conduit by the sodium in the alloy, and caustic soda and hydrogen will result. Through the conduit D flow to the chamber E caustic soda, impoverished alloy, and hydrogen. In the chamber E a separation occurs. Impoverished alloy or lead will return from the chamber E by the conduits F to the interior of the main furnace A, where it will again act as a cathode and take up sodium. From the chamber E the hydrogen will escape through the pipe I into the vessel J. Thence will also flow molten caustic soda. From the vessel J the hydrogen will pass

through the pipe K and will be consumed in the auxiliary melting-furnace L, where the salt is melted for maintaining the supply of salt in the main furnace A.

As already indicated, the contents of the furnace may at any time be removed through the pipe H.

Chlorin gas will escape through the conduit P and pipe  $p$ .

The contents of the furnace A are to be maintained molten and fluid. The temperature necessary to secure this condition will preferably be maintained by employing a suitable electric-current density on the anodes and cathode with a higher electromotive force than would ordinarily be required to decompose the molten salt. In other words, if reliance is placed upon the current to make up the loss of heat due to radiation and conduction the electrical energy will have to be in excess of that needed for the decomposition of the molten salt only. The heat resulting from the combination of the sodium with the oxygen of the steam will compensate to a considerable extent for loss of temperature incident to radiation and conduction. This is possible, because of causing the oxidation of sodium in close proximity to the molten electrolyte or causing the impoverished alloy or lead at the somewhat-higher temperature resulting from the reaction to immediately return and come in contact with or proximity to the molten electrolyte, so as to impart some of its heat thereto, thus reducing the amount of electrical energy required to be converted into heat energy to maintain the electrolyte in the molten state and making possible the operation of the furnace at a lower voltage and at a higher efficiency than would otherwise be the case. Burning the resultant hydrogen in the auxiliary furnace will obviously to some extent still further reduce the heat energy, which otherwise would have to be supplied from an external source or from a higher electromotive force of current. There may of course be some other and additional external source of heat. Thus an auxiliary supply of fuel-gas may be drawn through a pipe V and be consumed in the small melting-furnace together with or independently of the hydrogen. Caustic alkali formed at the high temperature contemplated will be practically anhydrous. It will be seen that there is a circulation continuously in the same direction through an endless circuit, that such circuit comprises two branches, one of said branches being the hearth of the main furnace A, containing that part of the molten metal in contact with the electrolyte, and the other the conduit D and its connections, that steam is introduced into the circulation, that alkali metal will be oxidized by the steam during transit, and that in such circuit or circulation one or more anodes are arranged.

It is more advantageous to cause the necessary circulation by steam than by mechanical devices, for the latter involve moving



parts requiring attention, and this advantage is independent of the advantage due to the utilization of the heat of combination during circulation.

5 Turning now to Figs. 4, 5, and 6, A designates a furnace like the one already described. It may have a cover and appurtenances of like construction similarly sealed. At its bottom is a hearth  $\alpha'$  and beneath it a  
 10 conduit D, communicating with the interior of the furnace near one end, as in the example of my invention already described. In this modification (illustrated by Figs. 4, 5, and 6) the conduit D terminates in a well D' at  
 15 that end which is farthest away from its communication with the furnace A. It will be observed that the bottom of the conduit D is inclined toward the well, or, in other words, it is arranged at an angle. It will also be  
 20 noticed that the well is at a lower level. A pipe H, like that already described, communicates with the bottom of the well and may form the negative terminal. Above the well is a chamber E, communicating by conduits  
 25 F with the interior of the furnace at the opposite end from that where the furnace is in communication with the conduit D. Through a diaphragm forming the bottom of the chamber and the top of the well D' vertically extends a conduit Q, which is open at both ends.  
 30 This diaphragm is shown as made of a removable piece and may be made of iron or steel. A steam-pipe G, having a controlling-cock  $g$  and preferably made of iron or steel, extends  
 35 downwardly through the cover of the chamber E and thence through the conduit Q nearly to the bottom of the latter. This conduit Q may be made of iron or steel. At the lower  
 40 end of the steam-pipe is a distributor R, which, as here shown, consists of a cap secured, by means of a screw-thread, to the lower end of the steam-pipe, so that there will be a space between its bottom and the lower  
 45 end of the steam-pipe. In this cap are a number of passages  $r$ , which communicate with its interior below the steam-pipe and extend through the upper end of the cap. Steam passing down through the steam-pipe will escape through the passages  $r$  in an upward direction.  
 50 In this modification the circulation takes place from the furnace A into the upper end of the conduit D, thence down into the well D', thence through the conduit Q into the chamber E, and thence into the furnace A. Steam will escape in an upward direction into the conduit Q and will thereby  
 55 elevate the alloy and oxidize the sodium in transit. Hydrogen and caustic soda pass through a pipe I, which communicates with pipes I' I<sup>2</sup>. The caustic soda flows through the pipe I' into a drum U, and the hydrogen passes off into an auxiliary melting-furnace similar to that shown in Fig. 1 or any suitable receiver, or it may be permitted to escape  
 60 into the air. This modification has advantages in that it affords easy access to parts of the apparatus sometimes requiring inspection,

for inspection may be had by removing the body of salt and then taking off the cover of the chamber E.

In both examples of my invention it will be understood that the decomposition of the steam and the formation of caustic soda and hydrogen will occur in transit.

I do not wish to be confined to producing caustic soda and chlorine by my invention, as I may employ the invention for the production of some other caustic alkali and gas.

What I claim as new, and desire to secure by Letters Patent, is—

1. In the manufacture of fused caustic alkali the process which consists in electrolytically decomposing a molten salt of alkali metal while resting on a body of molten lead constituting a cathode, and thereby forming an alloy of the lead and the alkali metal, circulating the molten body of cathode metal and alloy past an anode or anodes toward another molten body of lead or of lead and alloy, and introducing steam into the last-named molten body below its surface.

2. In the manufacture of fused caustic alkali the process which consists in electrolytically decomposing a molten salt of alkali metal while resting on a body of molten lead constituting a cathode, and thereby forming an alloy of the lead and the alkali metal, circulating the molten body of cathode metal and alloy past an anode or anodes toward another molten body of lead or of lead and alloy, and introducing steam into the last-named molten body below its surface in such manner as to effect the circulation.

3. In the manufacture of fused caustic alkali the process which consists in electrolytically decomposing a molten salt of alkali metal while resting on a body of molten lead constituting a cathode, and thereby forming an alloy of the lead and the alkali metal, circulating the molten body of cathode metal and alloy past an anode or anodes toward another molten body of lead or of lead and alloy, introducing steam into the last-named molten body below its surface, circulating the second-mentioned molten body of lead or of lead and alloy to a place where hydrogen and molten alkali will separate from the lead or alloy and removing the hydrogen and the molten alkali from the circulation.

4. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing a molten salt of an alkali metal while resting upon a body of molten lead constituting the cathode, and thereby forming an alloy of lead and the alkali metal, and introducing steam under pressure into a separate body of the alloy in fluid communication with the cathode to give a movement sufficient to cause the return of the lead or impoverished alloy to a point where it will again take up alkali metal, substantially as specified.

5. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing a molten salt of an alkali metal while resting upon a body of molten lead constituting the cathode, and thereby forming an alloy of lead and the alkali metal, and introducing steam under pressure into a separate body of the alloy in fluid communication with the cathode to give a movement sufficient to cause the return of the lead or impoverished alloy to a point where it will again take up alkali metal, substantially as specified.



- ically decomposing a molten salt of an alkali metal while resting upon a body of molten lead constituting the cathode, and thereby forming an alloy of lead and the alkali metal, 5 introducing steam into a separate body of the alloy in fluid communication with the cathode, and returning the resultant lead or impoverished alloy to a point where it will again take up alkali metal, substantially as specified.
- 10 6. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing a molten salt of an alkali metal while resting upon a body of molten lead constituting the cathode, and thereby 15 forming an alloy of lead and the alkali metal, introducing steam into the alloy, and burning the resulting hydrogen for supplying heat for the operation, substantially as specified.
- 20 7. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing a molten salt of an alkali metal while resting upon a body of molten lead constituting a cathode, and thereby forming an alloy of lead and the alkali metal, in- 25 troducing steam into the alloy, and burning the resulting hydrogen in contact with the salt required in the process and thereby melting such salt, substantially as specified.
- 30 8. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing in a suitable furnace a molten salt of an alkali metal while resting on a

body of molten lead constituting the cathode, and thereby forming an alloy of lead and the alkali metal, utilizing the heat of combina- 35 tion of the alkali metal with oxygen to conserve the heat energy of the process by injecting a proper quantity of steam under pressure through a separate body of the alloy in fluid communication with the cathode metal, 40 and returning the resultant lead or impoverished alloy at a higher temperature to the point where it again takes up the alkali metal.

9. In the manufacture of fused caustic alkali, the process which consists in electrolytically decomposing in a suitable furnace, a mol- 45 ten salt of an alkali metal while resting on a body of molten lead constituting the cathode, and thereby forming an alloy of lead and the alkali metal, utilizing the heat of combina- 50 tion of alkali metal with oxygen to conserve the heat energy by injecting a proper quantity of steam under pressure through a separate body of the alloy in fluid communication with the cathode metal and in close proximity 55 thereto, whereby the additional heat created will be directly conducted to the furnace.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

CHARLES ERNEST ACKER.

Witnesses:

W. LAIRD GOLDSBOROUGH,  
J. M. RIEMANN.