

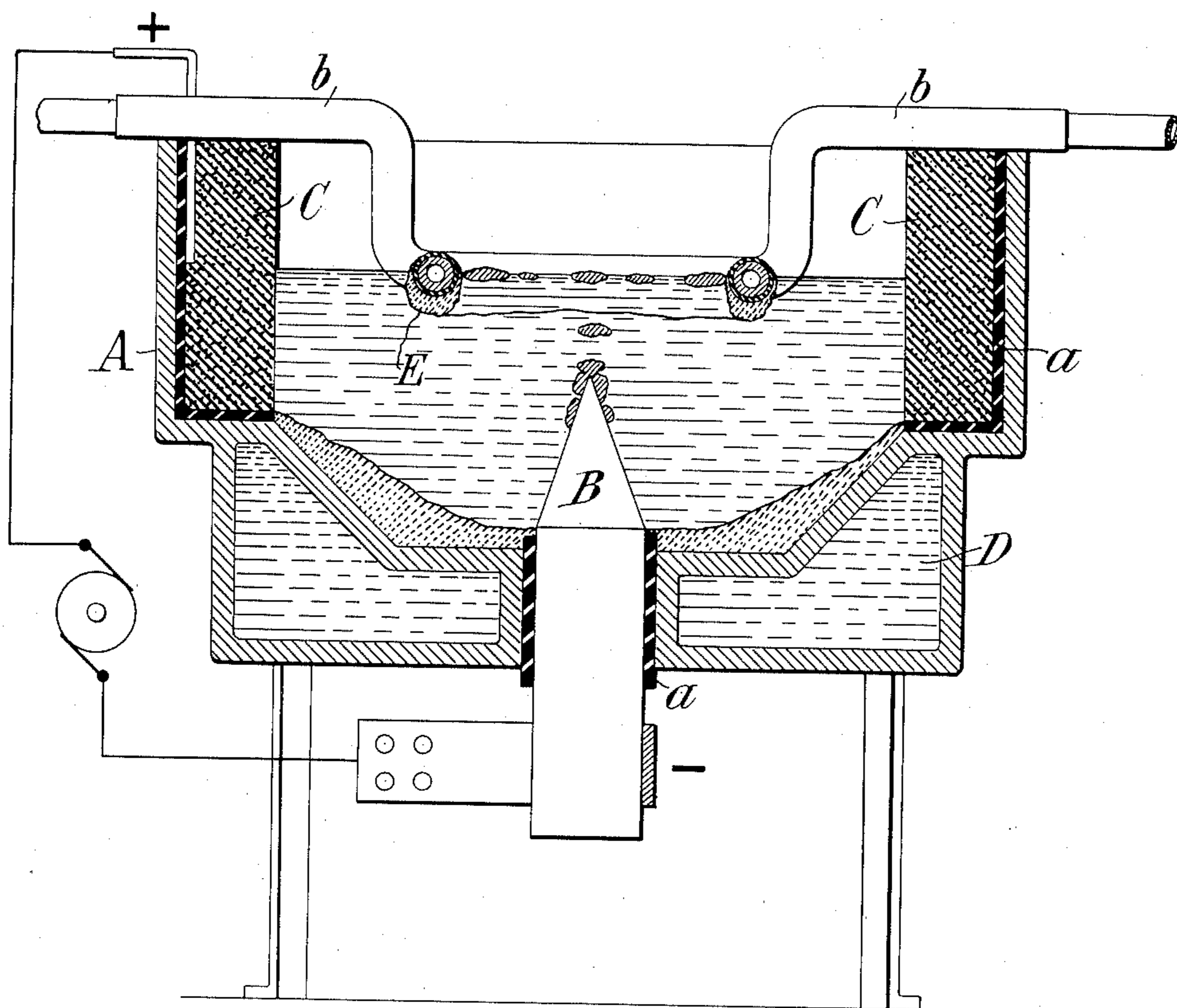
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PATENTED SEPT. 3, 1907.

G. O. SEWARD & F. VON KÜGELGEN.

ELECTROLYTIC PRODUCTION OF EARTH ALKALI METALS.

APPLICATION FILED APR. 25, 1906.



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# UNITED STATES PATENT OFFICE.

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TO VIRGINIA LABORATORY COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW  
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## ELECTROLYTIC PRODUCTION OF EARTH-ALKALI METALS.

No. 864,928.

Specification of Letters Patent.

Patented Sept. 3, 1907.

Application filed April 25, 1906. Serial No. 313,572.

*To all whom it may concern:*

Be it known that we, GEORGE O. SEWARD, a citizen of the United States, and FRANZ VON KÜGELGEN, a subject of the German Emperor, both residing at Holcombs Rock, in the county of Bedford and State of Virginia, have jointly invented certain new and useful Improvements in the Electrolytic Production of Earth-Alkali Metals, of which the following is a specification.

This invention relates to the production of earth-alkali metals and alloys thereof by the electrolysis of their fused salts. Calcium is a typical example of such metals; it has long been known that to produce calcium successfully by the electrolysis of fused calcium chlorid it was necessary (1) to maintain a high current density at the cathode, and (2) to maintain as low a temperature as possible in the electrolyte.

By the use of a submerged cathode, and by maintaining the temperature at the cathode so low that the metal did not melt together into globules, calcium has been produced in spongy form which was removed from time to time, being squeezed to expel the surplus electrolyte, after which the sponge was remelted. This method did not prove satisfactory, as the sponge is voluminous and must be removed frequently, and the remelting presents difficulties. Another method which has been practiced to some extent involves the employment of a cathode which just touches the surface of the electrolyte, so that the separated calcium adheres to the cathode, the latter being gradually lifted as the metal forms, so that the calcium is obtained in the form of a rod or stick. This method demands exceedingly close attention and much practical skill.

The present invention overcomes the objections hitherto encountered, and provides a method of producing calcium and similar metals and alloys which is characterized by simplicity of operation and may be practiced on a large scale.

By our invention the metal is separated at a high temperature in a molten state, and then removed quickly from the hot zone immediately surrounding the cathode and into a cooler portion of the electrolyte, thereby so reducing its temperature that there is little or no tendency for it to redissolve in the electrolyte or to burn in the air. Hitherto when the metal has been produced in a molten state at the cathode, it has remained in the hot zone, which, being at the top of the electrolyte, subjects the metal to the dangers of redissolution in the electrolyte and of burning in the air.

To facilitate an understanding of the process we refer to the accompanying drawing showing in vertical section a suitable form of apparatus.

The cell is formed by a vessel A, which may be of cast-iron and which may advantageously be of circular form, having a cathode B projecting up centrally

through its bottom, and an anode C of carbon or graphite arranged annularly around its sides and preferably concentric with the cathode. The cathode and anode are separated from the iron vessel by layers *a a* of suitable insulating material such as asbestos. The vessel is so designed or adapted that when filled to a suitable level with the salt or salts forming the electrolyte, and receiving a sufficient current to maintain the latter molten, the bottom and exposed portions of the vessel will be protected by a chilled layer of the electrolyte. This may conveniently be accomplished by forming the vessel with a water-jacket D through which cold water may be circulated. The cathode may be made of iron or steel, and is preferably tapered or conical, and terminates a suitable distance below the surface of the electrolyte. Above, and preferably concentric with the cathode, is arranged a collecting ring or partition E, which should be provided with means for water-cooling it so as to chill upon its surface a sufficient layer of the electrolyte to protect it. This cooling is best performed by circulating water through pipes *b b* which conduct it to and from the tubular ring forming the partition or collector.

In applying our invention to the production of calcium, the vessel is filled with fused calcium chlorid (with or without the addition of other salts) to about the level shown, so that the cathode is completely submerged and the collecting ring dips a little way into the electrolyte. The current density at the cathode is such that the calcium is formed in molten globules. These globules, when they become large enough, detach themselves from the cathode and float to the surface of the electrolyte, where they collect within the confining ring and fuse or cohere into a mass of constantly increasing volume and of sufficient firmness to be readily removed as desired.

The electrolyte above the cathode is much lower in temperature than the zone immediately surrounding the cathode, so that the metal in passing from the hotter portion rises through the cooler zone so that on reaching the surface it is cooled to such an extent that it does not burn by contact with the air. By increasing the depth of the electrolyte above the cathode this cooling of the metal may be increased, but it is preferable that the calcium should be soft enough when it reaches the surface to cohere with the previously formed metal in a uniform mass. The cooling effect of the air still further lowers the temperature of the metal, which may become quite solid in its upper part.

From time to time fresh calcium chlorid, preferably in a molten and completely dehydrated state, is added to replace that which is decomposed, the process being entirely continuous. Chlorin is of course liberated at the anode, and may be collected and recovered.

Although the arrangement of the apparatus has been described with some detail, yet it will be understood that the particular construction and proportions shown are not essential to our invention, and may readily be varied by those skilled in the art. For example, the water-cooled collecting ring may be varied in shape or may be replaced by any suitable material which is un-attacked by the electrolyte. The cooling of this ring is not indispensable, and it may be even possible to omit the ring altogether. Other shapes and arrangements of the anode and cathode may be used. The process is not limited to the use of a single cathode and a single anode, a plurality of these parts being admissible. The shape of the collecting ring may vary according to the shape and arrangement of the cathode and anode.

The invention is applicable not only to the production of calcium magnesium and other earth-alkali metals, but is also applicable to the production of such alloys of these metals as are sufficiently light to float to the surface of the electrolyte.

We claim as our invention the following defined improvements in the art of producing earth-alkali metals or alloys thereof by electrolysis of their fused salts of greater specific gravity than the metals or alloys produced, namely:—

1. Maintaining the current density at a submerged cathode so high that the metal is formed in a molten state, and cooling the metal which separates from the cathode while it is passing through the electrolyte.

2. Maintaining the current density at a submerged cathode so high that the metal is formed in a molten state, maintaining above the cathode a cooler portion of the electrolyte, and causing the metal to separate from the cathode and rise therethrough, whereby it is reduced in temperature.

3. Maintaining the current density at a submerged cathode so high that the metal is formed in a molten state, causing the metal to separate from the cathode and rise through a cooler portion of the electrolyte, whereby its temperature is reduced, and accumulating the metal within a collector.

4. Maintaining the current density at a submerged cathode so high that the metal is formed in a molten state, causing the metal to separate from the cathode and rise through a cooler portion of the electrolyte, whereby its temperature is reduced, and accumulating the metal within a water-cooled collector.

5. Maintaining a current density at a submerged cathode so high that the metal is formed in a molten state, and cooling the metal while rising from the cathode through a cooler portion of the electrolyte, sufficiently to prevent the metal burning in contact with the air, while keeping it sufficiently soft to cause it to form a coherent mass.

6. Producing an earth-alkali metal by electrolyzing a fused salt thereof with a submerged cathode, maintaining at the cathode a current density so high that the earth alkali metal is formed in a molten state and ascends therefrom, maintaining a cooler portion of the electrolyte above, through which the earth-alkali metal passes, whereby its temperature is reduced, and accumulating the same within a suitable collector.

7. Producing calcium by electrolyzing a fused calcium salt with a submerged cathode, maintaining at the cathode a current density so high that the calcium is formed in a molten state and ascends therefrom, maintaining a cooler portion of the electrolyte above, through which the calcium passes, whereby its temperature is reduced, and accumulating the same within a suitable collector.

In witness whereof, we have hereunto signed our names in the presence of two subscribing witnesses.

GEORGE O. SEWARD.  
FRANZ VON KÜGELGEN.

Witnesses:

J. H. WEBB,  
P. O. HARDING.