

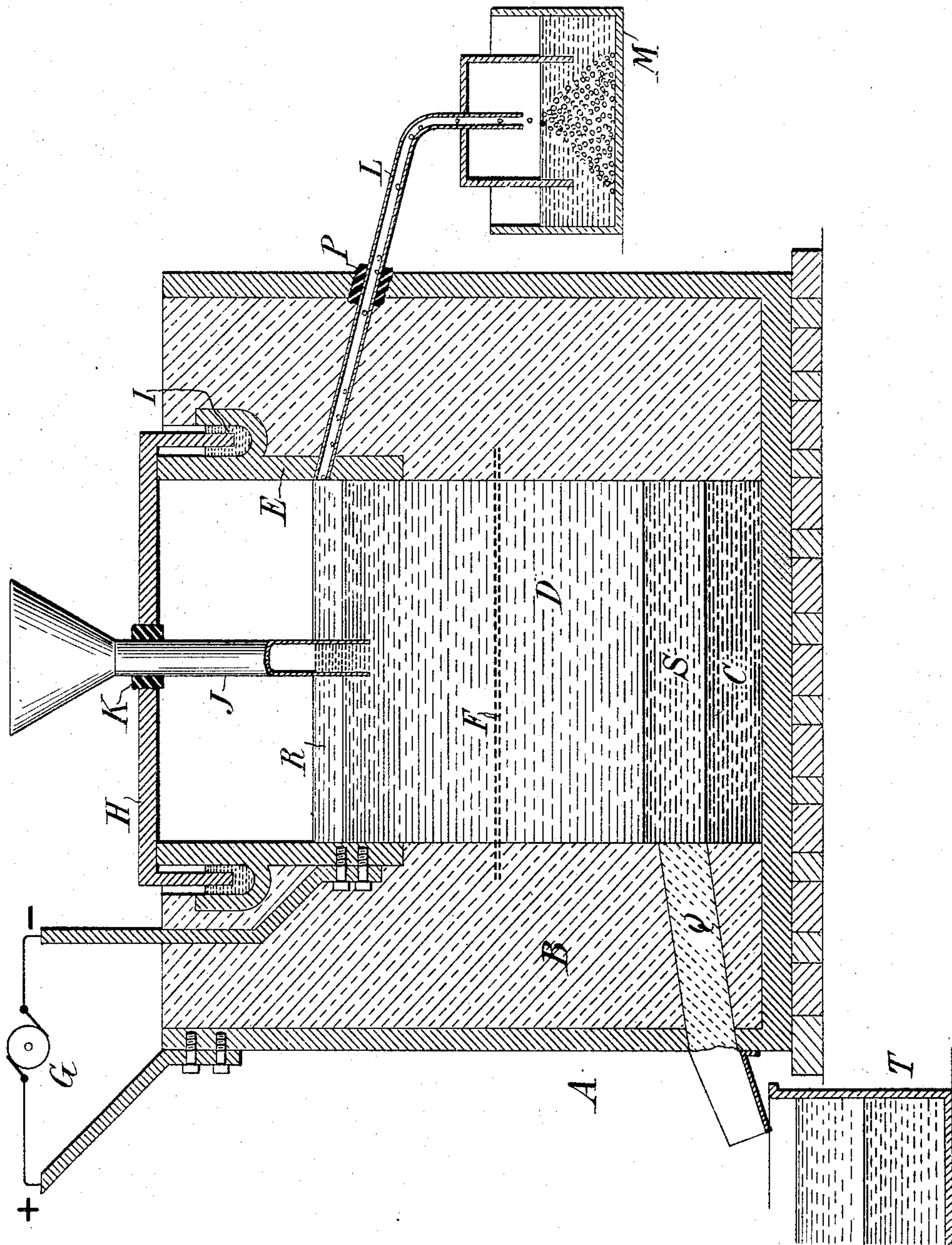
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F. VON KUGELGEN & G. O. SEWARD.

## PROCESS OF PRODUCING LIGHT METALS BY ELECTROLYSIS.

APPLICATION FILED MAY 4, 1905.



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# UNITED STATES PATENT OFFICE.

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## PROCESS OF PRODUCING LIGHT METALS BY ELECTROLYSIS.

No. 850,376.

Specification of Letters Patent.

Patented April 16, 1907.

Application filed May 4, 1905. Serial No. 258,821.

*To all whom it may concern:*

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

This invention relates to the production of metals of low specific gravity by the electrolysis of their fused salts.

It has been proposed to produce sodium by electrolyzing fused sodium chlorid, using a molten anode of a heavy metal, as silver or copper, so that the chlorin liberated at the anode forms a chlorid of the heavy metal. The chlorid thus formed was expected to remain at the bottom of the electrolyte, because of its greater specific gravity, and thus be kept separate from the sodium, which was separated at the cathode, which was located at the upper part of the electrolyte. This method appears at first to operate in accordance with theory, but after a little time the heavy metal is deposited at the cathode instead of sodium, and the output per unit of current becomes so low as to be unprofitable. On account of these difficulties the process has never been practiced commercially.

Our invention avoids the difficulties heretofore encountered in the effort to electrolyze fused salts of light metals by use of a soluble anode. By a "soluble" anode we mean one which is attacked by the chlorin or other electronegative element which is liberated at the anode to form a chlorid or other salt of the heavy anode metal. We have found that if such heavy chlorid of the anode metal can be kept from rising up to or near the cathode the separation of the heavy metal at the cathode, with its accompanying losses, can be avoided.

Our invention therefore involves the electrolysis of a haloid or other salt of a light metal by use of a soluble anode beneath of molten heavy metal and a cathode above, maintaining such conditions as to prevent the salt of the heavy metal from circulating upward through the electrolyte far enough to come into contact with the cathode or so nearly adjacent thereto as to be decomposed and separate the heavy metal of the cathode.

In the electrolysis of a haloid salt with a soluble anode of heavy metal the haloid salt of the heavy metal which is formed has a specific gravity sufficiently greater than that of the electrolyte, so that if undisturbed it would maintain itself in a separate layer between the electrolyte and the molten anode; but it has always been found that in electrolyzing with a current of sufficient density to produce the sodium there is such disturbance of the heavy salt as to cause it to mix with the lighter salt forming the electrolyte, with the result that the heavy salt is decomposed, thus liberating the heavy metal at the cathode, so that it recombines with the positive element or light metal and eventually stops the production of the latter.

In practicing our invention we operate under different conditions from those heretofore maintained, whereby we prevent the contact of the salt of the anode metal with the cathode. To this end we locate the cathode at the upper part of the electrolyte as remote as practicable from the heavy anode and avoid the formation of such currents as will carry the salt of the anode metal up into contact with the cathode, removing this salt from time to time as it forms, so as to keep its level as remote as possible from the cathode. We also interpose a diaphragm of foraminous metal, such as iron-wire cloth, between the cathode and the salt of the anode metal, whereby the latter is confined beneath such diaphragm. The use of such a metal diaphragm is rendered possible by the absence of free halogen or other electronegative agents, which would otherwise corrode and destroy the metal. The use of a diaphragm, however, is not under all conditions indispensable. It is important to construct the cell of a sufficiently large diameter in proportion to the current used so that the electrolytic action shall be diffused within the cell, so as to avoid the formation of energetic currents of liquid which tend to displace and carry up the salt of the heavy anode metal. In addition to these precautions it is of advantage to provide for the periodical settling of the heavy salt beneath the electrolyte, which may be done by discontinuing the electrolytic current at intervals, whereupon the heavy salt settles quickly to the bottom of the electrolyte. This precipitation of the heavy salt instead of being ef-



fects in the electrolytic cell may be accomplished in a separate settling-vessel by drawing off the electrolyte either intermittently or continuously into a vessel, wherein it remains so nearly quiescent as to permit the precipitation of the heavy salt, whereupon the light salt can be decanted or drawn off and returned to the cell.

We will proceed to explain the practical application of our process as applied to the production of sodium by electrolyzing sodium chlorid with a molten lead anode. In so doing we will refer to the accompanying drawing, which is a vertical section of an electrolytic cell, showing one suitable manner of constructing the cell for practicing our invention, it being understood, however, that various other mechanical arrangements and constructions may be substituted.

Referring to the drawing, let A designate an iron pot or vessel having a lining B, of fire-clay, magnesia, or other suitable material, and a pool of molten lead C or other heavy metal having a suitable affinity with the electropositive element to be liberated at the anode, so that it may serve as a soluble anode. Above this anode floats a body of the molten salt to be electrolyzed D, which in this instance is sodium chlorid, and at the upper part of the electrolyte it comes in contact with a cathode E, which may be of cast-iron, being shown as annular, forming a part of the outer wall of the cell. Between the anode and the cathode is extended in substantially horizontal direction a diaphragm F, which may consist of one or more layers of iron-wire cloth or may be made of any suitable material. Suitable electrical connections are made between the vessel A and the positive terminal of a dynamo or other electric generator G and between the negative terminal thereof and the cathode E. The cell is covered by a cover H, of iron, having an outer flange which is hermetically sealed, as by dipping into an annular trough I, containing lead which is maintained molten by the heat of the cell. Through this cover projects a feed-pipe J for introducing fresh electrolyte, its lower end being sealed in the electrolyte. This pipe J should be insulated from the cover by a packing of asbestos or other material K. For discharging the sodium a tube L leads out from the upper part of the cell and discharges into oil in a vessel M, whereby contact with the atmosphere is avoided. The tube L requires to be insulated from the vessel A by a packing P, of asbestos or other material. A tap-hole Q is provided for discharging the salt of the anode metal.

For practicing the process a suitable quantity of lead is first placed in the bottom of the cell to form the anode C, and the sodium chlorid to form the electrolyte is then introduced, preferably in the molten state, so that

its heat fuses the lead. The electrolyte is filled in to about the level shown, so that it covers the lower part of the annular cathode E. The cover H is applied to exclude air. The current being turned on, electrolytic action takes place, sodium is liberated at the cathode and floats on the electrolyte, as shown at R, and chlorine is liberated at the anode and attacks the lead, forming lead chlorin, which being of greater specific gravity than the electrolyte tends to remain in a separate layer beneath it, as shown at S. The internal currents induced in the electrolyte by the electrolysis tend to disturb this layer of heavy salt and carry it up through and mix it with the electrolyte. Such lifting of the lead chlorid or admixture thereof with the electrolyte as should bring a material quantity of it in contact with the cathode would defeat the process. This result we prevent by some or all of the following expedients: by the interposition of the diaphragm F, which prevents the dissemination of the heavy salt in the upper portion of the electrolyte above the diaphragm and retains the lead chlorid or mixture of lead chlorid and sodium chlorid entirely beneath the diaphragm; by confining the cathode to the upper part of the electrolyte and separating it as far as is practical from the lead anode, the cell being of such dimensions as to make this distance as great as practicable without prohibitively increasing the internal resistance; by tapping off the lead chlorid at sufficiently frequent intervals in order to maintain as wide a separation as practicable between its normal level and the cathode; by avoiding too great a current density, and thereby so energetic and localized an electrolytic action as to cause too active disturbing currents within the electrolyte, to which end the cell is made of large diameter for a given amperage, and by precipitating the lead chlorid out of the electrolyte whenever its admixture therewith becomes sufficient to disturb the operation or seriously diminish the efficiency, which is best done by discontinuing the current for a short time to enable the heavy salt to settle, or by opening the tap-hole Q the cell may be emptied of the lead chlorid and sodium chlorid and refilled with fresh electrolyte introduced through the pipe J. In such case the chlorids that are drawn off may be left in a settling vessel until the lead chlorid has been precipitated, whereupon the sodium chlorid may be drawn off or decanted and used as fresh electrolyte. If the two salts are not thus drawn off, the lead chlorid gradually accumulates as the electrolysis proceeds, so that its level rises, and to keep it as remote as possible from the cathode it is drawn off at intervals through the tap-hole Q. Fresh electrolyte is added at intervals through the pipe J. The lead cathode requires to be replenished occasionally.



Our process is useful not only in case of sodium, but also in the electrolysis of other metallic chlorids. The process is generally applicable to the production of any metal sufficiently light to float upon its salt used as the electrolyte. The anode must be a metal materially heavier than this electrolyte and having an affinity for chlorine or other electronegative element, so as to take up this element and prevent its interfering with the process by an independent reaction. Our process is chiefly useful for the electrolysis of chlorids, but may also be used with other haloid salts.

15 We claim as our invention—

1. The process of producing light metals from their salts, which consists in electrolyzing the fused salt in the presence of a soluble anode of heavy metal beneath, and a cathode  
20 above, thereby forming a salt of the heavy metal overlying the anode, and remote from the cathode, and preventing circulation of said heavy-metal salt upward through the electrolyte, whereby to avoid contact of such  
25 salt with the cathode.

2. The process of producing light metals from their salts, which consists in electrolyzing the fused salts in the presence of a soluble anode of heavy metal beneath, and a  
30 cathode above, thereby forming a salt of the heavy metal overlying the anode, and dividing the electrolyte by a diaphragm between such salt and the cathode whereby to prevent circulation and confine such salt to the lower  
35 part of the cell to prevent its reaching the cathode.

3. The process of producing light metals from their salts, which consists in electrolyzing the fused salt in the presence of a soluble

anode of heavy metal beneath and a cathode  
40 above, whereby is formed a salt of the heavy metal, and periodically permitting the precipitation of the heavy-metal salt which has become mixed with the electrolyte.

4. The process of producing light metals  
45 from their salts, which consists in electrolyzing the fused salt in the presence of a soluble anode of heavy metal beneath and a cathode above, whereby is formed a salt of the heavy metal, and discontinuing the electrolysis at  
50 intervals to permit the precipitation of the heavy-metal salt which has become mixed with the electrolyte.

5. The process of producing light metals from their salts, which consists in electrolyz-  
55 ing the fused salt in the presence of a soluble anode of heavy metal beneath, and a cathode above, thereby forming a salt of the heavy metal, and drawing off the latter salt periodically to keep its level remote from the cath-  
60 ode.

6. The process of producing light metals from their chlorids, which consists in electrolyzing the fused chlorid in presence of a  
65 soluble anode of heavy metal beneath, and a cathode above, with a horizontal metal diaphragm dividing the electrolyte, and periodically drawing off the salt of the heavy anode metal beneath said diaphragm, and adding  
70 fresh electrolyte above the diaphragm.

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

FRANZ VON KÜGELGEN.  
GEORGE O. SEWARD.

Witnesses:

J. H. WEBB,  
C. OFFERHAUS.