

J. WHITING.  
METHOD OF DECOMPOSING SALTS.  
APPLICATION FILED DEC. 16, 1908.

951,228.

Patented Mar. 8, 1910.

3 SHEETS—SHEET 1.

Fig. 1.

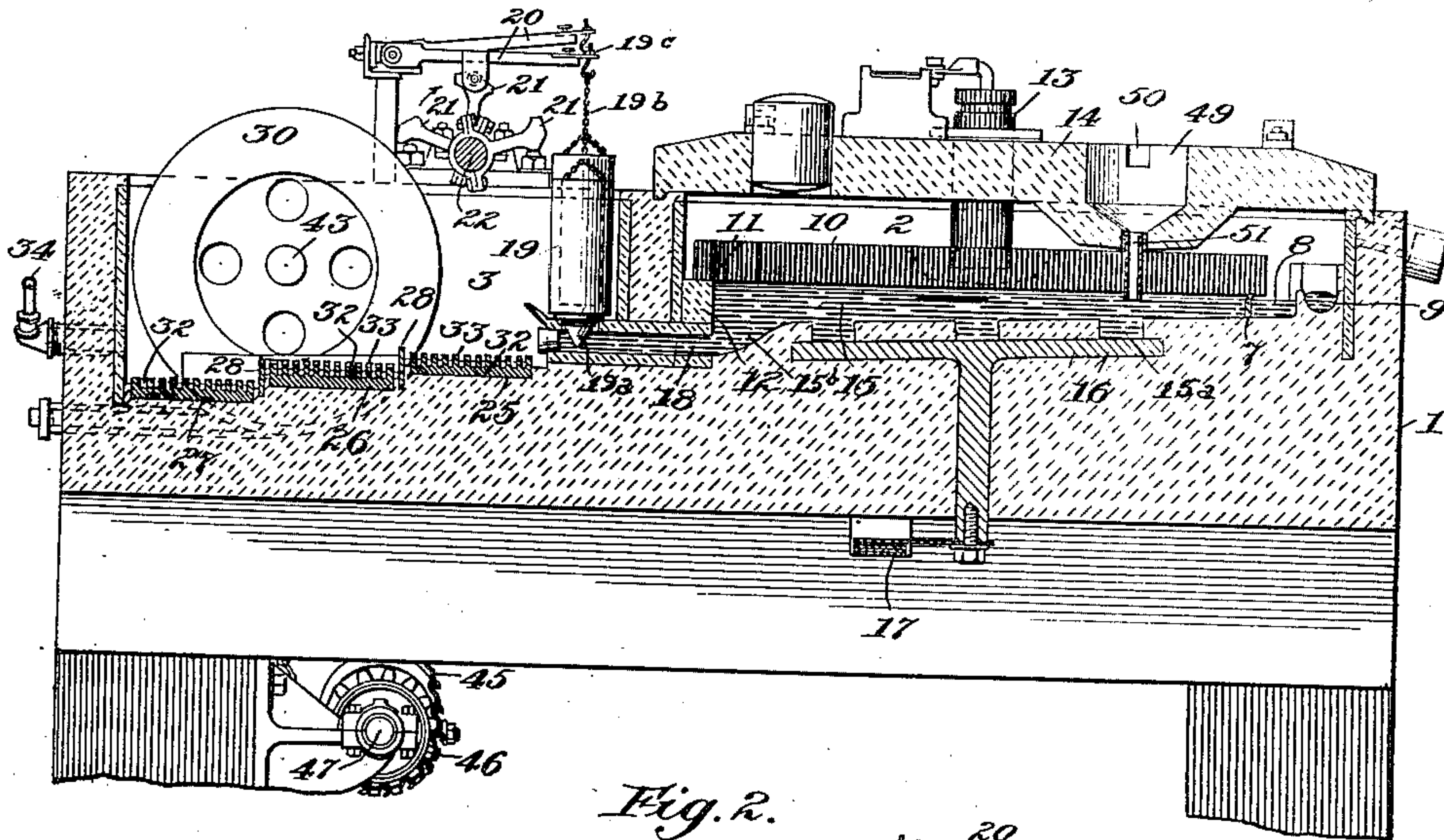
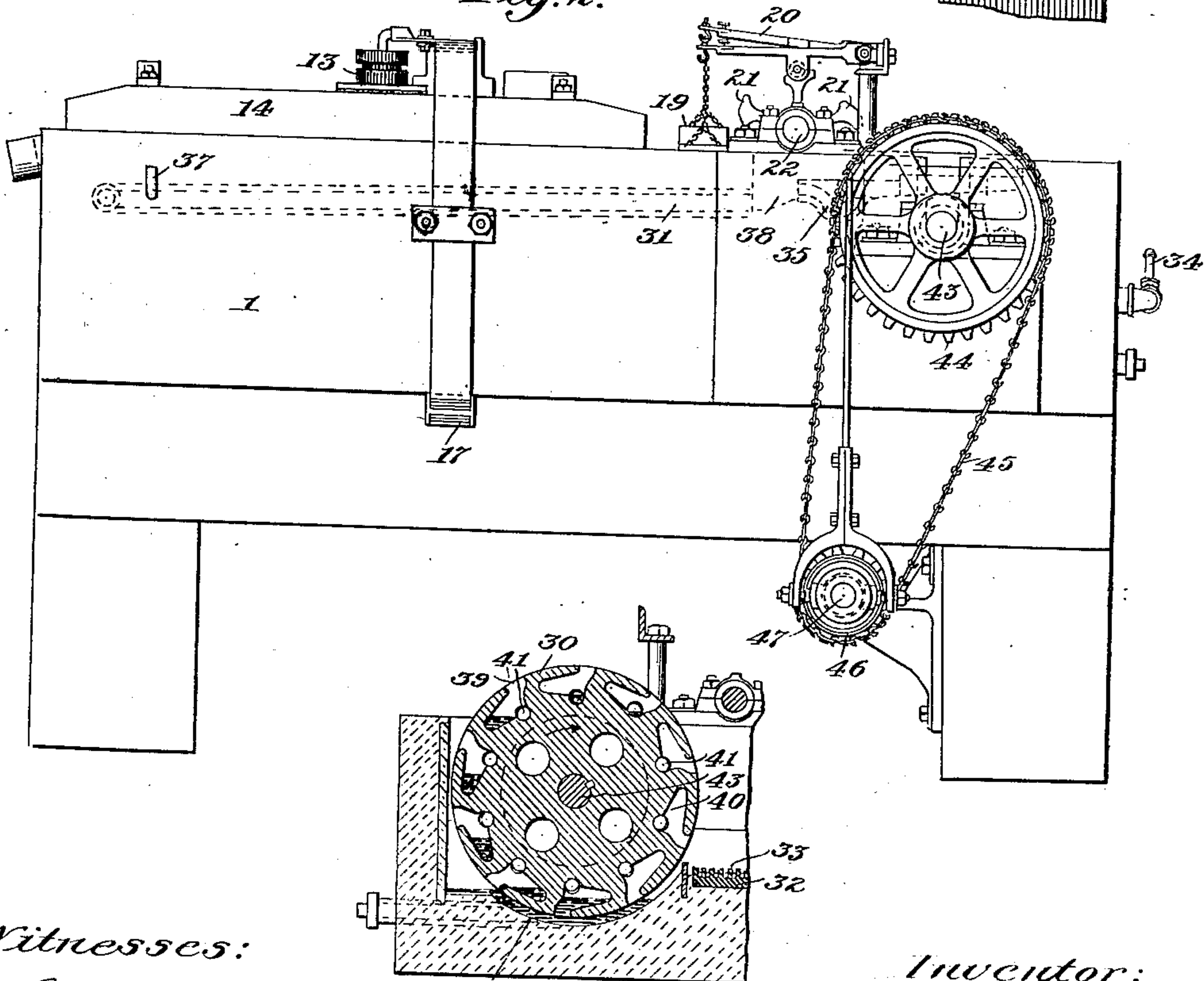


Fig. 2.



Witnesses:

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Chas. H. Potter.

Inventor:

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Fig. 5.

Byrne, Townsend, Beckett & Tamm,  
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3 SHEETS—SHEET 2.

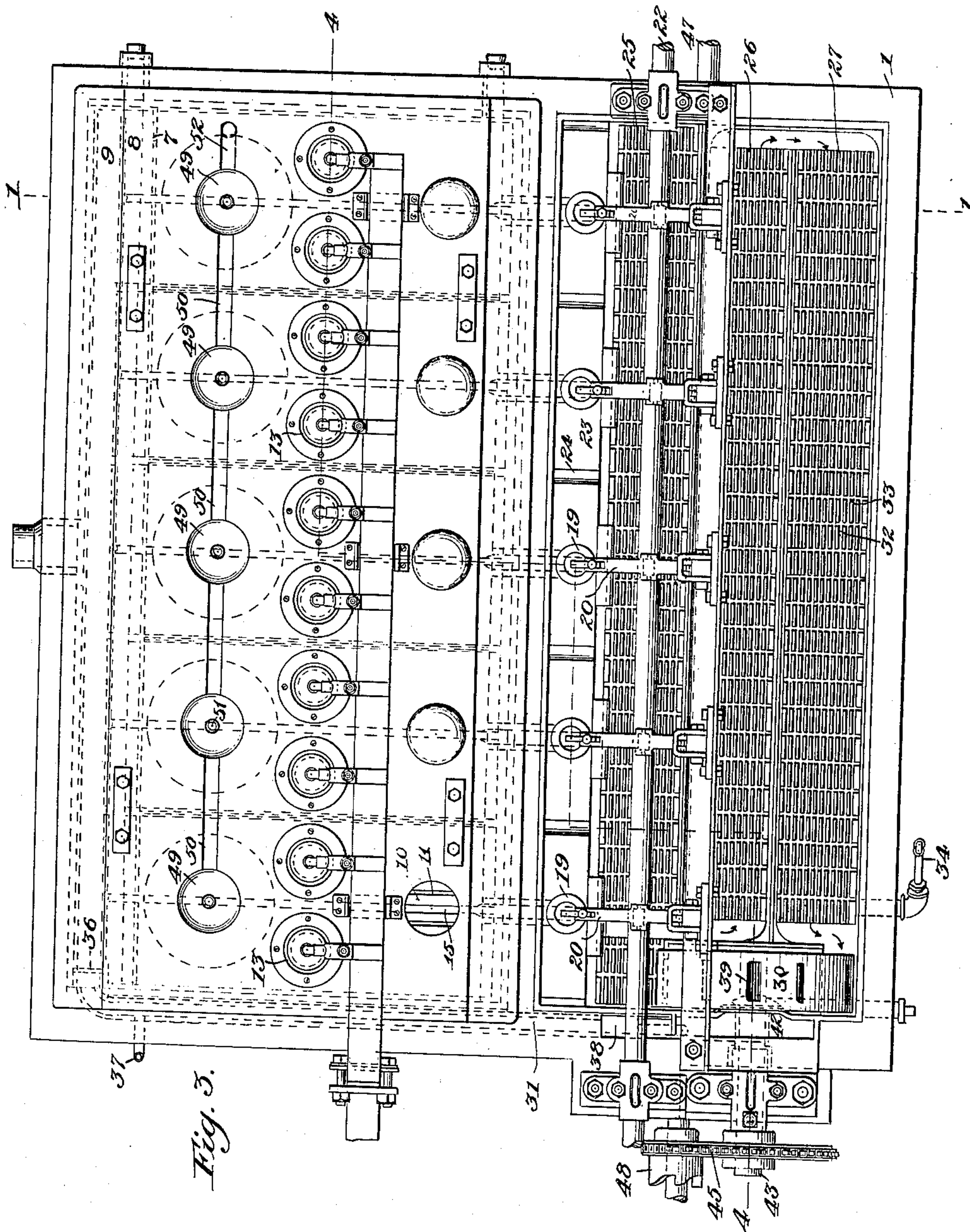


Fig. 3.

Witnesses:

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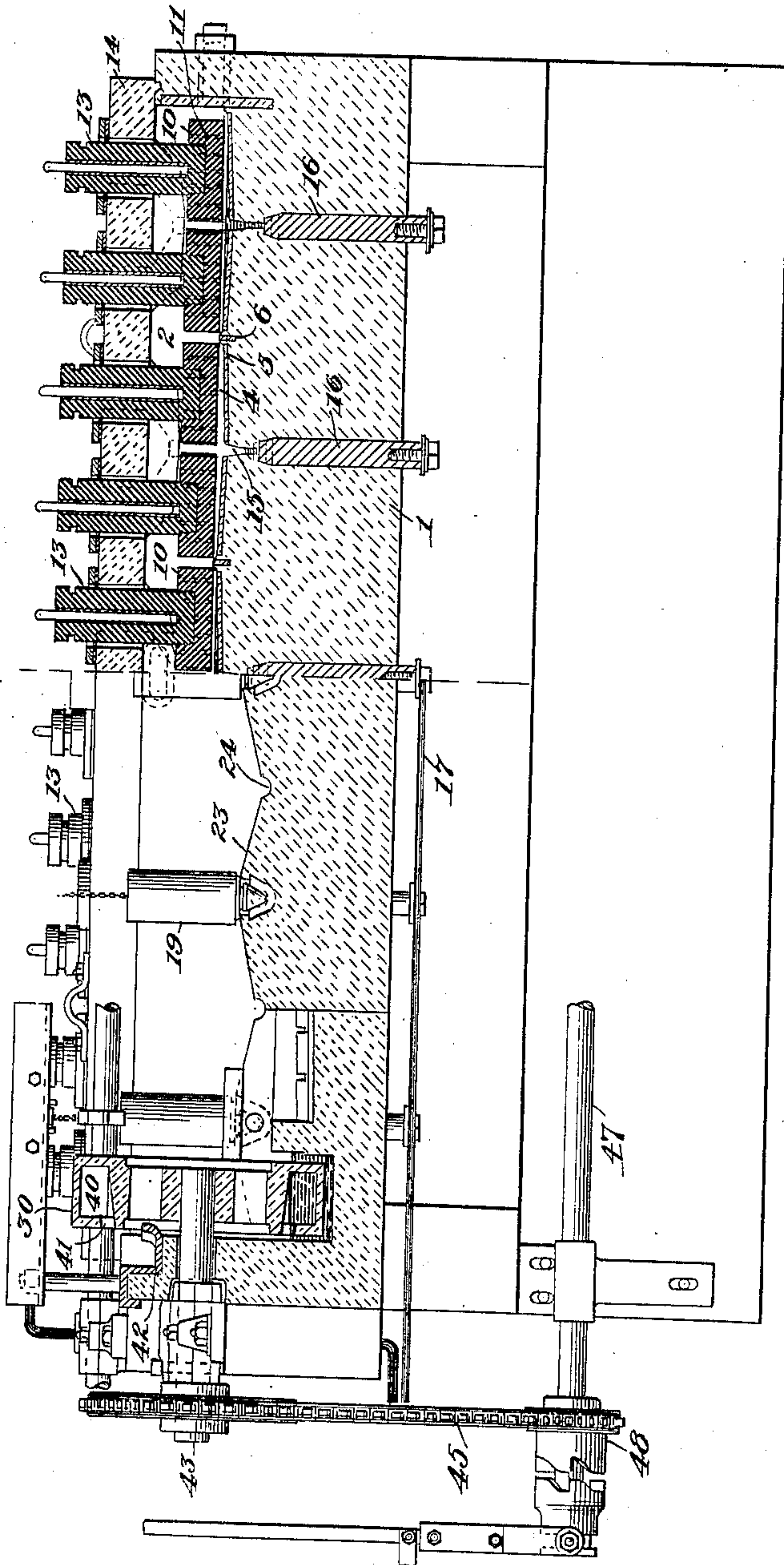
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3 SHEETS—SHEET 3.

Fig. 4.



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# UNITED STATES PATENT OFFICE.

JASPER WHITING, OF BOSTON, MASSACHUSETTS, ASSIGNOR TO THE WHITING COMPANY, OF BOSTON, MASSACHUSETTS, A CORPORATION OF MAINE.

## METHOD OF DECOMPOSING SALTS.

951,228.

Specification of Letters Patent.

Patented Mar. 8, 1910.

Application filed December 16, 1908. Serial No. 467,846.

*To all whom it may concern:*

Be it known that I, JASPER WHITING, a citizen of the United States, residing at Boston, in the county of Suffolk and State of Massachusetts, have invented certain new and useful Improvements in Methods of Decomposing Salts, of which the following is a specification.

This invention relates to the electrolytic decomposition of salts by methods involving the employment as cathode of a body of liquid metal or alloy. In methods of this character it has been usual heretofore to maintain a substantially continuous circulation of the cathode, but this practice has proven objectionable by reason of the tendency of the electrolytically separated metal, as sodium, to become oxidized and to reënter into solution in the decomposing compartment. For example, in the production of caustic soda by electrolysis of aqueous solutions of sodium chlorid in presence of a mercury cathode, the sodium amalgam tends to collect at the surface of the mercury cathode and its removal to the oxidizing compartment is only incompletely effected by the flow of the underlying body of mercury; the sodium of the amalgam is therefore subject to oxidation by the electrolyte undergoing decomposition, resulting in a reduced efficiency of operation.

In my prior patent No. 877,537, granted January 28, 1908, I have described a method of electrolyzing salts wherein a cathode of liquid metal or alloy is maintained in a quiescent state during the period of electrodeposition therein of an alkali or other metal, the cathode being periodically conveyed from the decomposing compartment to a suitable oxidizing compartment, and returned to said decomposing compartment, the flow of the electric current being preferably maintained without interruption by the provision of a plurality of bodies of the cathode metal adapted to be conveyed in succession to the oxidizing compartment or compartments. As described in said patent, the contents of the decomposing and oxidizing compartments are in balanced relation, and the metal or alloy which is discharged

periodically from the decomposing compartment is conveyed to a pump or equivalent device by means of which it is elevated and discharged into the oxidizing compartment, the denuded metal flowing thence by gravity to the decomposing compartment where its content of alkali metal is restored.

According to the present invention the liquid metal is also maintained in a state of rest during the charging period or period during which the sodium or other metal is deposited therein, but is at appropriate intervals transferred by gravity to a region of oxidation, the denuded or purified metal being thereafter returned to the decomposing compartment. In the preferred embodiment of the invention the alloy or amalgam from the decomposing compartment is permitted to flow through the oxidizing compartment in contact with carbon surfaces and an oxidizing electrolyte. By this procedure several important advantages are secured as hereinafter set forth.

For a full understanding of the invention the same will be described by reference to the production of caustic soda by the electrolytic decomposition of an aqueous solution of sodium chlorid in the presence of a cathode of mercury.

A preferred form of apparatus is illustrated in the accompanying drawing wherein,—

Figure 1 is a longitudinal section on line 1—1 of Fig. 3; Fig. 2 is a side elevation of the apparatus; Fig. 3 is a plan view of the same; Fig. 4 is a transverse vertical section on the broken line 4—4 of Fig. 3; and Fig. 5 is a vertical sectional view of a preferred form of elevating device or pump for the mercury.

Referring to the drawings 1 represents a body of cement, concrete or other suitable material, in which are formed one or more decomposing compartments 2 and an oxidizing compartment 3. As illustrated, I preferably provide a plurality of mechanically separated bodies of mercury contained in shallow basins 4, shown as five in number, formed in the bottom of the decomposing compartment 2: it will be understood how—



ever that a single body of mercury or any desired number of bodies may be provided in the decomposing compartment. These basins may be lined with glass, slate or earthenware 5 and are separated from each other by low partitions 6 set in the cement base and extending above the normal surface of the mercury cathode, the arrangement providing a common body of electrolyte above the several separated bodies of mercury. 7 is a similar partition disposed transversely of the basins 4 and near one end thereof in such manner as to provide an equalizing channel 8 from which the mercury may flow to the several basins, this equalizing channel being supplied with mercury from a distributing conduit 9 which receives the denuded metal from the oxidizing compartment.

Above each basin is disposed an anode or anodes 10, which may be vertically slotted as indicated at 11 to permit the escape of chlorine. These anodes are usually of graphite and are carried at one end by a ledge 12 integral with the cell body, and at the other by the partition 7. The anodes preferably extend over the mercury in the equalizing channel as shown in such manner as to constitute the mercury therein a part of the active cathode, whereby the capacity of the cell is increased to a corresponding extent. The several anodes are electrically connected in circuit through graphite leads 13 extending upwardly through the cover 14 of the decomposing compartment. The cover 14 is removable together with the anodes, this arrangement permitting the ready opening of the cell for inspection, cleaning or the repair or replacement of parts.

Centrally disposed in the bottom of each basin 4 is a longitudinal slot or channel 15, the mercury in which is in electrical contact with suitable conductors 16 which are embedded in the body 1 and are connected to the cathode bus bars 17. The slots 15 incline downwardly to the discharge end of the cell, and communicate at their lower ends with conduits 18 through which the amalgam may be discharged into the oxidizing compartment 3. At the bottom of each slot are shown a plurality of wells 15<sup>a</sup> adapted to retain small volumes of mercury which serve to protect the metallic conducting plates 16 and to establish electrical contact between them and the overlying amalgam. Each slot terminates at its lower end in a pocket 15<sup>b</sup>, the construction being such as to permit the mercury to be discharged from the cell or compartment with substantial completeness. Each conduit 18 is provided at its discharge end with a weighted valve 19 normally closing the same but adapted to be opened at desired intervals

by the movement of levers 20, actuated by cams 21, carried by a rotary shaft 22. The valves 19 are guided to their seats by downward projections 19<sup>a</sup>. As illustrated, they are suspended by chains 19<sup>b</sup>, and an adjusting screw 19<sup>c</sup> is provided by means of which the lift of the valve may be varied as desired. It will be understood however that the invention is not restricted to the particular means illustrated for operating the valves at the desired or predetermined intervals. When the valves 19 are opened the mercury or amalgam in the respective basins of the decomposing compartment flows by gravity upon the inclined surfaces 23 (Figs. 3 and 4) and thence through channels 24 to the oxidizing compartment 3.

In the particular form of apparatus illustrated, the oxidizing compartment comprises a series of wide shallow channels 25, 26, 27 disposed at successively lower levels and separated by partitions 28 so arranged that the amalgam is directed in a tortuous path as indicated by the arrows through the several channels, the denuded mercury flowing finally to the sump 29 (Fig. 5). It is then elevated by a rotary disk pump 30 and returned by conduit 31 (Figs. 2, 3) to the distributing conduit 9, passing thence to the equalizing channel 8 and the proper basin of the decomposing compartment.

As indicated in Figs. 1 and 3 the bottom of each of the channels 25, 26, 27 of the oxidizing compartment is constituted by a carbon or graphite slab 32, having integral members 33 projecting upwardly into contact with the water or oxidizing electrolyte, the members 33 being disposed to permit a ready flow of the amalgam while providing a maximum contact therewith. The oxidizing compartment is supplied with water, the caustic soda solution escaping through an outflow 34.

It is important that the denuded mercury should be freed from adhering or mechanically conveyed caustic soda solution before reentering the decomposing compartment. In the present construction this may be accomplished by the provision of traps or seals of any ordinary type in the conduits 31 and 9. Thus I have illustrated in Fig. 2 a trap or seal 35 arranged in connection with the discharge line of the pump 30 and serving to retain the supernatant liquid while permitting the flow of the mercury. A similar arrangement comprising a partition extending below the surface of the mercury in distributing conduit 9 is indicated at 36 in Fig. 3. If desired the mercury may readily be washed and thereby thoroughly freed from alkaline constituents by the introduction of water into the conduit 31 at a point intermediate these seals, as at 37,



(Figs. 2, 3), this water overflowing at 38 into the oxidizing compartment 3 and serving as a source of supply therefor.

For elevating the denuded mercury from the sump 29 to the conduit 31 leading to the decomposing compartment, I have illustrated a rotary disk pump 30 provided with peripheral inlet apertures 39 leading to internal pockets 40 (Fig. 5), from which the mercury is discharged laterally through conduits 41 as the pockets are brought successively into proper relation to a trough 42, leading to the conduit 31. As shown, the pump 30 is mounted on a shaft 43 carrying a sprocket 44, driven by a chain 45 from a sprocket 46 on a shaft 47 having a clutch connection 48 with a power shaft. This apparatus for elevating the mercury is found to operate without such agitation of the metal as would result in its subdivision into globules with consequent danger of loss, and is claimed in my co-pending application, Ser. No. 467,848, filed December 16, 1908.

I prefer to provide in connection with the cell an intermittently operating device for supplying the electrolyte to the decomposing compartment, this device being automatic in character and permitting the electrolyte to flow into the several basins of the decomposing compartment only while the mercury or amalgam is absent therefrom or substantially so, that is to say, while the electrodeposition of sodium is not in progress, thereby avoiding all tendency to disturb the surface layer of amalgam and to reoxidize the deposited alkali metal. As herein illustrated this feed device comprises a plurality of receptacles 49 disposed in the cover of the cell above the respective basins 4, and interconnected by open channels 50. From the bottom of each receptacle 49 a tube 51 extends downwardly below the normal surface of the mercury in the basins 4 in such manner as to be sealed thereby. Electrolyte is supplied to one of the receptacles 49 and conveyed by the equalizing channels 50 to each of the others, any excess being permitted to return to the circulating system, or if desired to flow continuously into the decomposing compartment as through conduit 52.

So long as the several basins 4 contain sufficient mercury to seal the lower ends of the discharge tubes 51, the electrolyte is prevented from flowing therethrough, but when the mercury is discharged from any basin into the oxidizing compartment this seal is broken and a quantity of electrolyte corresponding approximately to the capacity of the receptacle 49 flows into the decomposing compartment. By this arrangement the fresh electrolyte is introduced at the bottom of the cell beneath the body of partially spent and consequently lighter elec-

trolyte therein, and distributes itself beneath the latter, thereby providing a body of substantially saturated electrolyte at the precise point where the electrolytic decomposition is most active. Furthermore by the arrangement described this introduction of electrolyte occurs while there is no amalgam in the cell, and is therefore without tendency to agitate the amalgam or to re-dissolve the alkali metal. The electrolyte may however be continuously fed to the decomposing compartment under any conditions which will not so agitate or disturb the body of electrolyte as to cause undue reoxidation of the alkali metal. The above described means for introducing electrolyte is claimed in my copending application, Ser. No. 467,847, filed December 16, 1908.

In the operation of the apparatus described the mercury in the several basins 4 beneath a common body of electrolyte in the decomposing compartment 2 remains in a quiescent state during the charging period, that is to say the period during which the sodium is being electrically deposited, and is therefore under the most favorable conditions as regards efficiency of operation. At the conclusion of the electrodepositing or charging period, when the mercury has received the desired quantity of sodium, the valve 19 controlling the outflow of one of the basins is raised by the corresponding cam 21, and the entire body of amalgam in this basin, with the exception of such small quantity as is required to maintain a seal in the conduit 18 between the decomposing and oxidizing compartments, flows rapidly by gravity but without serious agitation to the oxidizing compartment 3, traverses the same and is thereby denuded of its alkali metal. The pure mercury is then conveyed by pump 30 to the conduit 31 and thence through conduit 9 and equalizing channel 8 to the decomposing compartment, flowing as will be understood into an empty or discharged basin, in preference to one containing mercury. By the slow rotation of the shaft 22 the next cam 21 will after an appropriate interval be brought into position to raise the valve 19 of the basin next in series, the operation continuing in such manner that the mercury in the several basins 4 is discharged in rotation and at proper intervals, denuded of its alkali metal, and returned to the decomposing compartment. As the amalgam is discharged from each basin a body of fresh electrolyte is introduced through the tube 51 beneath the body of partially spent electrolyte therein, thereby maintaining the required degree of saturation of the electrolyte. While the liquid cathode is maintained during the charging period in a state of rest, it will be observed that during the



oxidizing period it is caused to flow in contact with carbon surfaces and an oxidizing electrolyte, thereby providing the most effective conditions for completely denuding it of its content of alkali metal.

The herein described method possesses several important advantages over that described in my prior patent, No. 877,537, above referred to, these advantages following chiefly from the mode of operation which permits the amalgam to flow by gravity from the decomposing to the oxidizing compartment instead of being elevated to the oxidizing compartment and being permitted to flow by gravity therefrom to the decomposing compartment. Among such advantages are the following:—(1) During the charging period the mercury and superposed electrolyte in the decomposing compartment is mechanically separated from that in the oxidizing compartment instead of being in balanced relation therewith, whereby it is not subject to fluctuation in level or variation in quantity due to the changes in the relative density of the electrolytes or in the pressure of the evolved gases: therefore more nearly constant current conditions may be maintained and the general operation of the cell improved. (2) There is less liability to the conveyance of caustic soda or other alkaline constituents from the oxidizing to the decomposing compartments, the flow of the denuded mercury to the decomposing compartment being tortuous, involving passage through a pump or elevating device and providing opportunities for further purifying operations to be performed upon it. (3) The arrangement permits the equalizing channel for the mercury to be placed within the decomposing compartment, thereby rendering the mercury in this channel available for use as a part of the cathode and correspondingly increasing the cell capacity. (4) The method provides for the pumping of pure mercury instead of amalgam, this being advantageous as avoiding all tendency to the formation of incrustations outside of the oxidizing chamber. (5) The construction is such as to permit the mercury to be discharged with substantial completeness from the decomposing cell or compartment. (6) The cell is more compact, more economical as regards the use of mercury, and of simpler and less expensive construction.

I claim:

1. The method of decomposing salts which consists in passing an electric current through an electrolyte to a quiescent body of liquid metal as cathode, transferring said body by gravity to a region of oxidation, and returning the denuded metal to the region of electrolytic decomposition.

2. The method of decomposing salts which consists in passing an electric current through an electrolyte to a plurality of quiescent bodies of liquid metal as cathode, transferring said bodies successively by gravity to a region of oxidation, and supplying denuded metal to the region of electrolytic decomposition.

3. The method of decomposing salts which consists in passing an electric current through an electrolyte to a quiescent body of liquid metal as cathode, transferring said body by gravity to a region of oxidation, imparting motion to said body during the oxidation, and supplying denuded metal to the region of electrolytic decomposition.

4. The method of decomposing salts which consists in passing an electric current through an electrolyte to a plurality of quiescent bodies of liquid metal as cathode, transferring said bodies successively by gravity to a common region of oxidation and supplying denuded metal to the region of electrolytic decomposition.

5. The method of decomposing salts which consists in passing an electric current through an electrolyte to a quiescent body of liquid metal as cathode, transferring said body by gravity to a region of oxidation, maintaining in said region of oxidation a continuous flow of liquid metal or alloy, and supplying denuded metal to the region of electrolytic decomposition.

6. The method of decomposing salts which consists in passing an electric current through an electrolyte to a quiescent body of liquid metal as cathode, transferring said body by gravity to a region of oxidation, elevating the denuded metal and returning it to the region of electrolytic decomposition.

7. The electrolytic method of decomposing alkali metal salts which consists in alternately charging a body of liquid metal with an alkali metal and freeing it therefrom, maintaining the liquid metal at rest during the charging period and causing it to flow in contact with a carbon surface and an electrolyte while freeing it from the alkali metal.

8. The cyclical electrolytic method of decomposing alkali metal salts which consists in alternately and repeatedly charging a body of liquid metal with an alkali metal and freeing it therefrom, maintaining the liquid metal at rest during the charging period and causing it to flow in contact with a carbon surface and an electrolyte while freeing it from the alkali metal.

9. The method of electrolyzing salts with a liquid metal cathode which consists in providing decomposing and oxidizing compartments containing independent bodies of liquid metal, passing an electric current to



a plurality of quiescent bodies of liquid metal in the decomposing compartment, transferring said bodies successively by gravity to the oxidizing compartment, and  
5 supplying denuded metal to the decomposing compartment.

10 10. The method of electrolyzing salts with a liquid metal cathode which consists in providing decomposing and oxidizing compartments containing electrically independent bodies of liquid metal, passing an electric current to the body of metal in the decom-

posing compartment, transferring said body by gravity to the oxidizing compartment, permitting it to flow therethrough in contact with a carbon surface and an oxidizing electrolyte, and returning the denuded metal to the decomposing compartment. 15

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

JASPER WHITING.

Witnesses:

JOHN G. PALFREY,  
BERTHA S. CHASE.