(No Model.)

A. J. ROGERS.

PROCESS OF AND APPARATUS FOR REDUCING METALS BY ELECTROLYSIS.

No. 296,357. Patented Apr. 8, 1884.

Fig. 1.

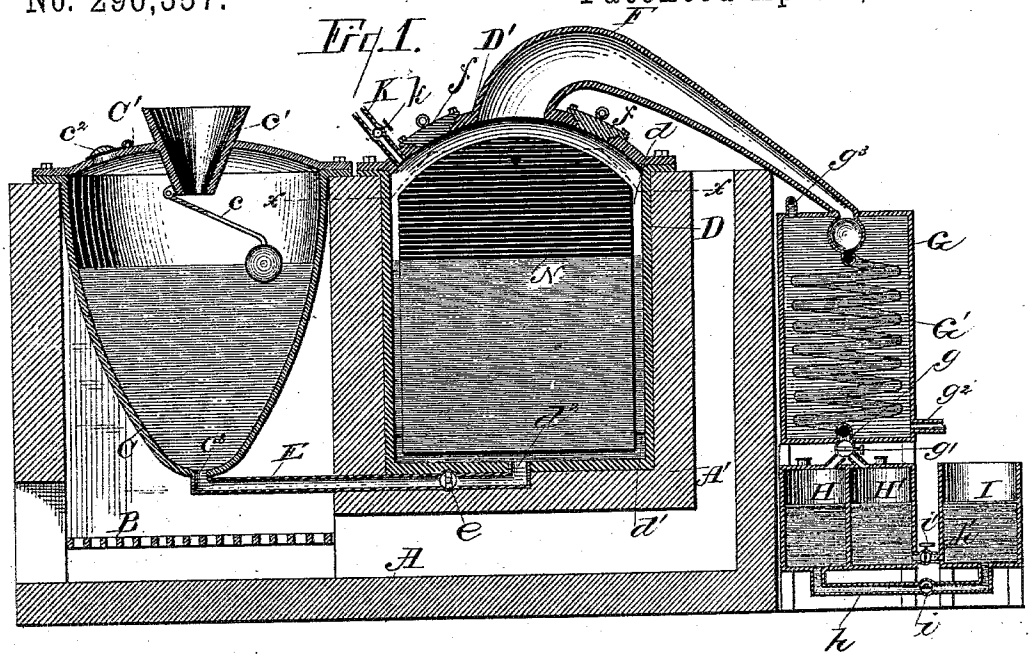
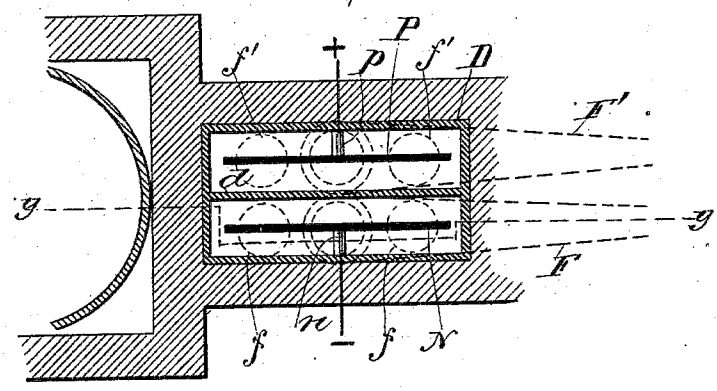


Fig. 2.



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UNITED STATES PATENT OFFICE.

AUGUSTUS J. ROGERS, OF MILWAUKEE, WIS., ASSIGNOR OF TWO-THIRDS TO HENRY MANN AND JULIUS KOEBIG, BOTH OF SAME PLACE.

PROCESS OF AND APPARATUS FOR REDUCING METALS BY ELECTROLYSIS.

SPECIFICATION forming part of Letters Patent No. 296,357, dated April 8, 1884.

Application filed April 21, 1883. (No model.)

To all whom it may concern:

Be it known that I, AUGUSTUS J. ROGERS, of the city of Milwaukee, in the county of Milwaukee and State of Wisconsin, have invented new and useful Improvements in the Process of and Apparatus for Reducing Metals and Metalloids by Electrolysis; and I do hereby declare that the following is a full, clear, and exact description thereof.

My process consists in heating the compound (meaning oxide, ore, or salt) or a mixture of compounds to the point of fusion, and submitting the "electrolyte" (so called) thus obtained to the action of the electric current. This passing of the electric current through the electrolyte is called "electrolysis." The result of the electrolysis would be that in general the metal would go to the negative electrode and the metalloid or acid to the positive electrode. This method of electrolysis is especially applicable to those elements having great affinity for oxygen—such as sodium, potassium, calcium, barium, strontium, magnesium, aluminium, boron, silicon, phosphorus, &c.—since they take oxygen readily from water or air; hence the electrolysis could not take place in an aqueous solution of the salt. In general, it is better to use the binary compounds—such as oxides, chlorides, bromides, iodides, fluorides, sulphides, &c.—but when a binary salt volatilizes easily after reaching the point of fusion, or offers great resistance to the electric current, ternary or other compounds may be used. It is also sometimes of advantage to use a mixture of compounds to increase the conductivity of the electrolyte or to lower the point of fusion—*e. g.*, the addition of calcium chloride to potassium chloride or to sodium chloride in equal molecular proportion lowers the point of fusion of potassium chloride from seven hundred and thirty degrees to about five hundred degrees. So, also, in the electrolysis of magnesium chloride, the addition of potassium chloride and ammonium chloride lowers the point of fusion,

In the accompanying drawings, which illustrate a convenient form of apparatus for carrying out my process, Figure 1 represents a vertical longitudinal section of the melting-pot, decomposing-tank, and condensing apparatus on the line *y y* of Fig. 2; and Fig. 2 is a horizontal section of a portion thereof, taken on the line *x x* of Fig. 1.

A A′ represents suitable masonry, within the walls of which my apparatus is to be located, and B is a fire-bed beneath the melting-pot C. The latter is preferably of the shape shown, in order that the products of combustion may encompass as much of its exterior surface as possible. The said pot is provided with a tight cover, C′, removably secured thereto, as shown, but furnished with a funnel or hopper, *c′*, in its center, adapted to be closed by a float-valve, *c*, when it is full—that is, so arranged that it will receive no further supply of salt when the liquid obtained by the melting of that first put into the pot rises as high as the lower projecting edge of the hopper *c′*. The cover C′ is further fitted with a safety-valve, c^2, to guard against danger from any sudden pressure occasioned by formation of gas.

D is the decomposing-tank, divided into two parts by the partition *d*, of porous material—such as fire-clay—and which extends down to within a short distance of the bottom of the tank, as indicated by the line *d′* in Fig. 1.

E is a pipe extending from a hole, d^2, in the center of the bottom of the tank D just beneath the partition *d*, to a hole, c^3, in the bottom of the melting-pot C, and this pipe is provided with a suitable stop-cock, *e*, to govern the passage of the melted material from the pot to the tank.

N and P are respectively negative and positive electrodes placed within the two sections of the decomposing-tank, and connected by wires (which are protected by passing through fire-clay or other tubes, *n* and *p*, against the corroding action of the electrolyte or its constituent) with the electric generator. (Not shown.)

The tank D is furnished with a tight-fitting cover, D′, which is perforated to receive two goose-necks, F F′, (one only, on the negative side, being shown in Fig. 1, but the position of both, as well as of all the man-holes, being represented by dotted circles in Fig. 2,) and man-holes *f f* and *f′ f′*. The central partition, *d*, of the tank D must fit gas-tight with or against the said cover, so that there may be no communication above the liquid in the

tank between the negative and positive sides thereof, the only communication between the two divisions of the tank for any free liquid or free gas being, therefore, by means of the liquid in the tank, at the bottom under the central partition, although the electric current, as well as the irons of the electrolyte, may pass freely through the partition *d* by reason of its porous character.

G is a condenser provided with a worm, G′, connecting at its upper end with the small end of the goose-neck F, and at its lower end having an outlet, *g*, guarded by a cock, *g′*, below which is a branched tube, the forks of which lead into different receptacles H H′, the cock being so constructed as to shut off communication between the condenser and either or both of the said receptacles at will, and the latter may either be rigidly connected together and to the other portions of my device, as shown, or may be each bodily removable, and they are provided with close-fitting covers, which may also have openings, suitably closed, therein, if desired.

I is an open-topped tank for containing liquid hydrocarbon, as hereinafter described, and *h* and *h′* are two pipes connecting the said tank with the receptacles H and H′, respectively, provided with suitable cocks, *i* and *i′*. The goose-neck F′ (indicated by dotted lines on the positive side) is not connected with a condenser ordinarily, but may lead off into any other suitable tank or receptacle, for the purpose hereinafter expressed.

My apparatus, thus described, is especially intended for the production of pure sodium and potassium from their salts, and by way of illustration I will describe its use in the reduction of chloride of sodium. The salt is poured into the melting-pot C (which is preferably of the shape shown, and may be made of clay, iron, or other heat-resisting non-corrosive material) through the funnel in the cover and subjected to such a degree of heat as will keep the contents with the constant new supply of sodium chloride some degrees above the point of fusion—say about 500° centigrade.

The decomposing-tank D is preferably made of clay or other similar heat-resisting material, which should be non-conducting, in the form of apparatus herein illustrated, and it should be protected from loss of heat by conduction or radiation, as by walling it in with the furnace, as here shown. The best shape for this tank is that of a rectangular parallelopiped, deep and narrow, and one which is one foot in width, three in length, and four in depth would be of convenient proportions for practical working purposes. Several tanks of this or other size could be used at one time by being connected in a series. Within the said tank, divided as hereinbefore described, are preferably located the electrodes N and P, which are most conveniently carbon plates, connected by proper wires with the electric generator, which latter may be a dynamo-machine, although the particular kind of generator is not material, that named being the best in ordinary cases, as the greatest amount of electric power may be most economically obtained thereby. These carbon plates should present as large a surface as practicable in the electrolyte, and should be near together to decrease the resistance to the passage of the current through the electrolyte, which latter consists of the melted sodium chloride conveyed into the decomposing-tank through the pipe E.

Into the condenser G cold water is constantly passed through the inlet g^2, which as it becomes heated passes out through the outlet g^3; and into the open tank I is poured a quantity of coal-oil or other suitable hydrocarbon in liquid form, which in turn passes through pipes *h* and *h′* into the receptacle H and H′, being normally at the same height and under the same pressure in all three of the vessels H H′ and I.

My device being constructed as described, and the melting-pot supplied with the salt and heat sufficient to fuse the same being applied, the salt is liquefied and run into the decomposing-tank, and the electric circuit closed, a current passes from the positive to the negative electrode, decomposing the electrolyte in such passage into sodium and chlorine, which collect, respectively, on the negative and positive electrodes. The temperature is such that the sodium is vaporized and passes up and out through the goose-neck F and through the worm G′ into one of the receptacles H or H′, (according to the way the cock *g′* is turned,) and then falls to the bottom of said tank, being specifically heavier than the coal-oil, which latter prevents any possible contact of the oxygen of the outside air with the sodium, which may be collected in its solid form from the said tank, as desired, while the chlorine is led, in its gaseous condition, up through the other goose-neck, F′, into any suitable receptacle, where it may be brought into contact with lime to form a bleaching-powder, or otherwise disposed of.

I have shown my electrodes as carbon plates suspended within the divisions of the decomposing-tank; but I do not confine myself to any exact arrangement of the apparatus in matters of detail, since it is obvious that my process could be carried out with success with my said apparatus modified in a variety of ways. For instance, one or both of the electrodes might be against the sides of the tank, or, indeed, (with proper outside insulation) might form the side or sides of the tank itself, or the carbon plates might project downward through the cover of the tank, or be arranged in any suitable way not inconsistent with the carrying out of my described process.

At any convenient point in my apparatus—say the cover of the decomposing-tank—I propose to place a small pipe, K, with suitable cock, *k*, the object of which is to afford means of connection with the flexible tube of an air-pump, so

as to exhaust the air from the interior of the decomposing-tank prior to the commencement of operation, after which, through this same pipe, (or from another point, if preferred,) hydrogen or other suitable gas is introduced into the upper part of the negative side of the tank, or into its goose-neck F, so that the passage which conducts the vaporized sodium (or potassium, if that substance is under production) into the receptacle H or H', will be supplied with said gas to prevent contact of oxygen with the contents of the said passage at the commencement of the process of operation.

The hydrogen gas might be used to force air out from the tank, by reason of its introduction therein, under pressure, without first having recourse to an air-pump, and it would be well to maintain communication between the upper part of the negative side of the decomposing-tank and a reservoir of the said gas during the entire process, under guard of a safety-valve, to guard against danger from rapid condensation or evolution of sodium vapor.

I am aware that heretofore sodium has been eliminated in an atmosphere containing no oxygen. I am also aware that various other salts than those which form the subject of my present invention have been decomposed by the aid of electricity after having been fused or melted; and I am also aware that it has been proposed to decompose the chlorides, iodides, or bromides of the metals of the alkalies or alkaline earths by electrolysis in a melting-pot or crucible, and that simultaneously gas freed from moisture and carbonic-acid gas has been introduced (in the treatment of double chloride of magnesium and potassium) in order to drive off the chlorine and exclude oxygen; but, so far as I am aware, my hereinbefore-described process, in its entirety, as pointed out in my claims, has never been applied to either the salts which I claim, nor to any other salts, nor has my particular apparatus been employed for such or any similar reductions, as I believe I am the first to separate sodium or potassium from sodium chloride or potassium chloride by first heating the described salts to the point of fusion in a suitable vessel; secondly, conveying the liquid thus obtained to a separate independent decomposing-tank provided with electrodes and passing an electric current therethrough; thirdly, conducting the gaseous chlorine and vaporized sodium or potassium into separate receptacles, that for the latter containing coal-oil, and the passage which conducts the vaporized sodium or potassium into its receptacle being supplied with hydrogen or other suitable gas. This is my process in its entirety, and I believe it to be new, as well as my apparatus designed for the continuous, successful, and practical working of said process in all its steps in the order named.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. In an apparatus for the reduction of metals and metalloids by electrolysis of their salts or ores in a melted condition, the combination of a melting-pot located over a suitable fire-bed, and an independent decomposing-tank provided with electrodes, and a close-fitting cover having gas-tight passages leading therefrom, and a condensing apparatus and receptacles, together with suitable connecting pipes and cocks, all arranged and adapted to operate substantially as set forth.

2. The process of separating sodium or potassium from sodium chloride or potassium chloride, substantially as described, consisting in heating the salt to the point of fusion in a suitable vessel, conveying the liquid thus obtained to a separate independent decomposing-tank provided with electrodes, and passing an electric current through the liquid to separate the chlorine therefrom, and then conducting the gaseous chlorine and vaporized sodium or potassium into separate receptacles, that for the latter containing coal-oil, and the passage which conducts the vaporized sodium or potassium into the said receptacle being supplied with hydrogen or other suitable gas, to prevent contact of oxygen with its contents at the commencement of the process of separation, substantially as set forth.

In testimony that I claim the foregoing I have hereunto set my hand, on this 17th day of April, A. D. 1883, in presence of two witnesses.

AUGUSTUS J. ROGERS.

Witnesses:
NETTIE H. VAN HORN,
RUDOLPH KAUFMANN.