(No Model.)

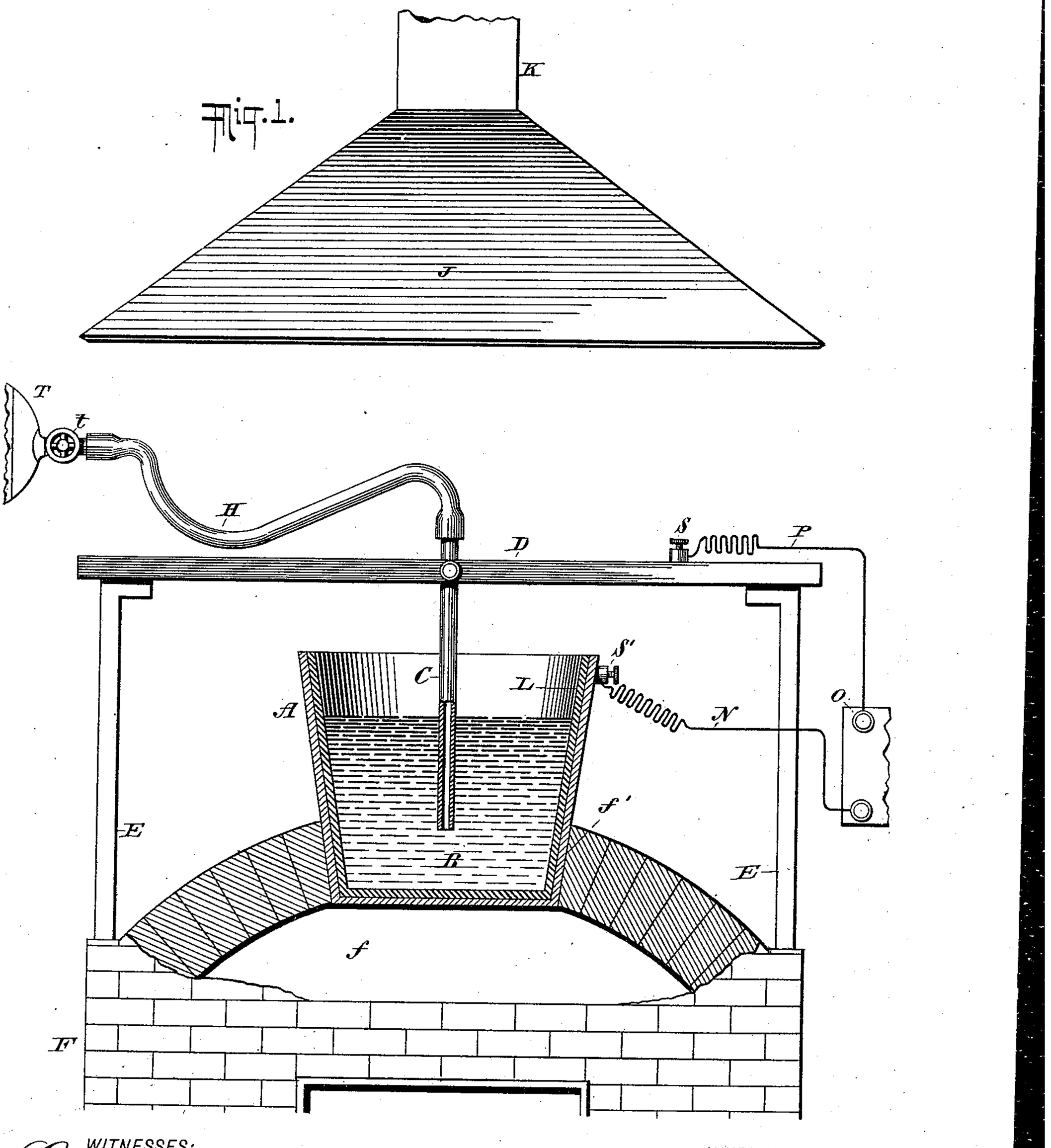
2 Sheets—Sheet 1.

L. WALDO & F. A. GOOCH.

PROCESS OF REDUCING ALUMINIUM.

No. 527,846. Patented Oct. 23, 1894.

Fig. 1.



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(No Model.) 2 Sheets—Sheet 2.

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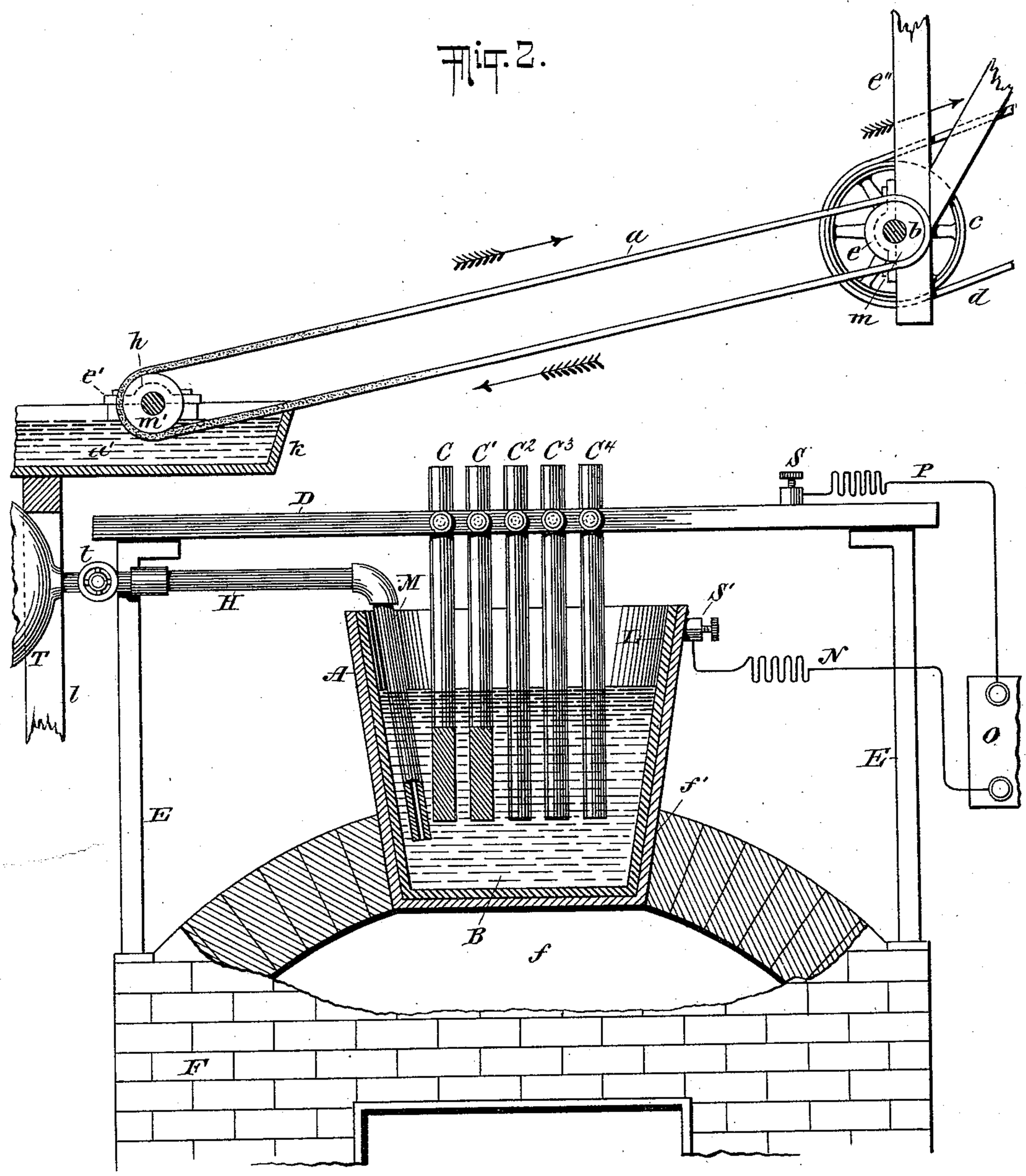
Fig. 2.



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UNITED STATES PATENT OFFICE.

LEONARD WALDO, OF BRIDGEPORT, AND FRANK A. GOOCH, OF NEW HAVEN, CONNECTICUT; SAID WALDO ASSIGNOR TO THE WALDO FOUNDRY, OF NEW JERSEY.

PROCESS OF REDUCING ALUMINIUM.

SPECIFICATION forming part of Letters Patent No. 527,846, dated October 23, 1894.

Original application filed September 1, 1893, Serial No. 484,561. Divided and this application filed March 23, 1894. Serial No. 504,763. (No specimens.)

To all whom it may concern:

Be it known that we, LEONARD WALDO, residing at Bridgeport, in the county of Fairfield, and FRANK A. GOOCH, residing at New Haven, in the county of New Haven, State of Connecticut, have invented certain new and useful Improvements in Processes of Reducing Aluminium; and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same, this being a divisional application of the application filed by us September 1, 1893, Serial No. 484,561.

The invention described herein relates to processes for the reduction of aluminium from its compounds by the electrolysis of a suitable fused mass containing such compounds.

In general terms, our invention comprises an improved process for reducing aluminium from its compounds, which consists in passing an electric current through a suitable fused bath containing a suitable halogen compound of aluminium, thereby electrolyzing the bath, and in introducing hydrogen into the bath. The compound of aluminium used should be one which will fuse at a temperature and under conditions which shall be practically attainable.

In practice we find that the halogen compounds of aluminium which are most suitable and best adapted to be used in our process are the chloride of aluminium and the fluoride of aluminium. By the introduction of hydrogen into the bath in the manner above described, a more uniform intermixture of the ingredients of the bath is secured. The surface of the molten mass is protected from atmospheric action when the hydrogen is used in some excess. The anodes are protected from the corrosive action of the halogens set free by the electrolysis of the bath, and such free halogens are converted into a more manageable form.

For convenience, we describe our invention with special reference to the reduction of aluminium from its chloride, it being understood, however, that our invention is also applicable to other compounds of aluminium.

In the accompanying drawings, which are hereby made a part of this specification, is shown one form of apparatus suitable to the practice of our invention. We do not, however, limit ourselves to the apparatus shown, since any other suitable apparatus would answer equally well for the practice of our invention.

Figure 1 represents a sectional diagrammatic view of the apparatus employed by us. Fig. 2 represents a sectional view of a modified form of apparatus employed by us.

Similar letters refer to similar parts throughout the drawings.

F is a furnace having a fire box *f*, and opening *f'* in the top of the furnace, into which is set a tapering crucible A, the same being supported by the sides of the opening *f'*.

The construction of the crucible and the arrangement of the electrodes and conductors are hereinafter fully described.

T is a tank from which the hydrogen is supplied through the tube H, the supply being regulated by the cock *t*. *s s'* are binding posts for the conductors N, P.

The construction and arrangement of other parts of the apparatus employed are hereinafter fully described.

In the practice of our invention, so far as it relates to the reduction of aluminium from its chloride, we preferably prepare a bath by fusing in a suitable tank or crucible A, and in proper proportions, the chloride of aluminium and the chloride of an alkaline metal, (preferably sodium,) or of an alkaline earth metal, with the fluoride of aluminium and the fluoride of sodium. A convenient way to form a suitable bath is to combine the chloride of aluminium and the chloride of magnesium with the mineral cryolite. We find it of advantage to add to the combination just named a certain proportion of chloride of sodium, which has the effect of making the bath more liquid. These ingredients are preferably mingled together in the fol-

lowing proportions: cryolite, fifty-five parts; chloride of aluminium, twelve parts; chloride of magnesium, five parts; chloride of sodium, twenty-eight parts; but the proportions above given are variable serviceably within considerable limits without materially affecting the operation or function of the bath, as, in fact, any proportions which may be found suitable may be employed,

The reason for employing cryolite or the fluorides of aluminium and sodium in the bath, is the well known fact that, when they are present and reduction by the action of the electric current takes place, the aluminium melts and flows to globules; while, when the bath contains only the chlorides named, the aluminium is obtained in the form of a powder, which has to be further treated before it can be made commercially available.

The crucible A, preferably employed by us in the operation of our invention, consists of a tank of iron lined with compacted carbon. The carbon lining L of this tank serves as the cathode, the iron of the tank being connected by the conductor N with the negative pole of a dynamo electric machine or other suitable source of electricity O. The anode C is a detached carbon electrode partially immersed in the bath B; and connected in the following manner with the source of electricity: We preferably suspend the anode from an iron bar D raised above the crucible and resting on wooden supports E E. The bar D is connected by the conductor P to the positive pole of the source of electricity.

If desired, the cathode may also be formed of a detached carbon, suspended in a similar way to that just described for the anode, or otherwise supported, and partially immersed in the bath B. If desired, a number of separate carbons may be used to form the electrodes as shown in Fig. 2, where C C′ C^2, C^3, and C^4, all suspended from the bar D, collectively form the anode. The relative superficial areas of the electrodes in any particular case are adjusted so as to secure the greatest economy in action. In place of a carbon anode, an anode of any other suitable material, as of platinum, may be used. The bath may be fused, and the fusion may be maintained by the application of heat from any suitable source, as from a furnace F. The temperature required to fuse our preferred bath is that of a dull red heat. We may also fuse the ingredients of the bath in a separate apparatus and introduce the same in a molten condition into the bath at the beginning of the process, the fusion being thereafter maintained in any suitable way. When the electric current is passed through the bath and electrolysis takes place, aluminium is deposited at the cathode and the halogens (chiefly chlorine) are liberated at the anode. It is well known that these gases, in a free state, are highly noxious to health. On this account, and for the further reason that they attack and quickly destroy the positive electrode in the apparatus used in any process similar to the one described, their escape in a free state would render such process commercially useless. To obviate these difficulties, and to accomplish other desirable results, we introduce into the bath hydrogen. The hydrogen is preferably delivered in a substantially continuous supply underneath the surface of the fused mass in near proximity to or through the anode. In the latter case, a longitudinally perforated carbon rod C is preferably employed for the anode, and the gas is passed through the same as through a pipe, the gas being supplied through a tube H connecting with the upper end of the anode C, which may project above the bar D. The hydrogen may also if preferred be conducted through a separate pipe as the pipe M in Fig. 2. When the halogen elements are liberated by the electrolysis of the bath, they combine instantly with the free hydrogen and escape at the anode in the form of hydrochloric acid or hydrofluoric acid, chiefly the former. These gases, in the quantities evolved in our process, are easily controlled and disposed of, without injury to apparatus or workmen, by appropriate means external to the bath, as by the use of a flue K, having a strong draft, and provided with a hood J, projecting over the crucible A; (see Fig. 1;) or cloths or other absorbent media, saturated with water, may be suspended over the crucible, the escaping gases being taken up by the water, whose power of acting as a solvent for hydrochloric acid and hydrofluoric acid is well known. Such an arrangement is shown in Fig. 2, where *a* represents an endless band or apron of cloth or other suitable fabric, passing over drums *m m′* attached to shafts *b*, *h*. The shaft *b* revolves in journal boxes *e* and is supported in any suitable manner, as by a strip *e″* suspended from the ceiling. The shaft *h* revolves in boxes *e′* formed on the edges of the trough *k*. The shafts and band or apron *a* are kept in revolution in the direction indicated by the arrows by power from any suitable source transmitted by the belt *d*, passing over the pulley *c*, which is rigidly attached to the shaft *b*. The shaft *b* is hung somewhat higher that the shaft *h*. The drum *m′* on shaft *h* runs close to or under the surface of water *w* contained in a shallow pan or trough *k*, having suitable support *l*. The band or apron *a* being inclined passes through the water *w* on the lower side of the drum *m′* and is thus kept saturated with water. Thus saturated it passes continuously over the crucible.

We do not claim the particular arrangement of devices just described, not regarding it as novel, nor do we confine ourselves to the particular devices shown, since any other suitable means external to the bath might be employed for disposing of the escaping gases.

We preferably supply hydrogen to the bath in excess of the amount required to combine

with the halogen elements. The excess of hydrogen substantially permeates the fused mass, thereby securing a more uniform intermixture of the ingredients of the bath and protecting the surface of the molten mass from atmospheric action.

In the practical operation of our invention, where we use the suggested bath composed of the chloride of aluminium and the chloride of magnesium and the fluorides of aluminium and sodium, with or without the chloride of sodium, we have found that we obtain at the anode hydrochloric acid almost exclusively, with very little trace of fluorine. This shows that the aluminium deposited at the cathode is derived chiefly from the chloride of aluminium in the bath; and we have found that, by the occasional addition of chloride of aluminium (Al_2Cl_6) in sufficient quantities, the bath will be replenished, and the process will be a substantially continuous one during a considerable period of time. An excess of the chloride of aluminium produces no injurious effect, as it is gradually utilized in the subsequent operation of the process.

We have hereinbefore spoken of fluorine being set free, though we are aware that it is considered very difficult to isolate this element. We, however, use the term "fluorine" merely for convenience, meaning thereby to include not only free fluorine, if any be liberated, but also any compound thereof, which might be set free at the anode, if hydrogen were not supplied to the bath in the process.

As before stated, the aluminium is deposited at the cathode, where it melts forming globules, which, when the suggested bath whose proportions are hereinbefore stated is employed, gather at the bottom of the bath, the aluminium having a higher specific gravity than the fused bath. The aluminium may then be removed in any suitable manner, as by means of a ladle lined and covered with carbon, or by drawing it off at the bottom through a suitable opening; or the bath may be poured out, and, after being cooled, the aluminium may be picked out.

In case a bath should be used whose specific gravity is higher than that of aluminium, the aluminium would rise to the top, where it could be easily removed.

In the process herein described, we use an electric current of suitably low electro-motive force, usually of from four to ten volts; but the voltage of the current employed will depend on the form and size of the electrodes as well as on the composition of the bath, it being only necessary to have a voltage sufficient to decompose electrolytically the compound of aluminium in the bath under all the existing conditions of resistance, and of chemical constitution.

The electric current which we employ in our process, and which is herein referred to as being passed through the bath, is employed solely for purposes of electrolysis. The hydrogen may in the first instance be introduced before or after the electric current is passed through the bath or simultaneously therewith, the order of operations being unimportant.

We are aware that hydrogen has been employed heretofore in processes for reducing aluminium, either as the sole agent or as an auxiliary agent in accomplishing the reduction of the metal from the aluminium compounds used. In the process herein described, we do not employ hydrogen as an agent in such reduction, nor do we in this application claim any process in which hydrogen is so employed. The objects of introducing hydrogen into the bath in our improved process herein described are, as before stated, to secure a more uniform intermixture of the ingredients of the bath, to protect the surface of the molten mass from atmospheric action, to protect the anodes from the corrosive action of the halogens set free by the electrolysis of the bath, and to convert such free halogens into a more manageable form.

We do not herein specifically claim those forms of the process herein described which are particularly set forth and claimed in our application filed September 1, 1893, Serial No. 484,561, of which this application is a division.

It being possible to form a number of different baths to any of which our invention would be applicable, we do not limit our invention to the use of any particular bath which has been described herein.

Having described our invention, what we claim, and desire to secure by Letters Patent, is—

1. As an improvement in the art of manufacturing aluminium, the herein described process, which consists in forming a bath by fusing the chloride of aluminium and the chloride of an alkaline earth metal with the fluoride of aluminium and the fluoride of sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and supplying hydrogen to the bath, substantially as and for the purposes set forth.

2. As an improvement in the art of manufacturing aluminium, the herein described process which consists in forming a bath by fusing together the chloride of aluminium and the chloride of magnesium with the fluorides of aluminium and sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and supplying hydrogen to the bath, substantially as and for the purposes set forth.

3. As an improvement in the art of manufacturing aluminium, the herein described process, which consists in forming a bath by fusing together the chloride of aluminium, the chloride of an alkaline earth metal and the chloride of sodium with the fluorides of aluminium and sodium, passing an electric current of suitably low voltage through the

fused mass, thereby electrolyzing the same, and supplying hydrogen to the bath, substantially as and for the purposes set forth.

4. As an improvement in the art of manufacturing aluminium, the herein described process, which consists in forming a bath by fusing together the chlorides of aluminium, magnesium and sodium and the fluorides of aluminium and sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and supplying hydrogen to the bath, substantially as and for the purposes set forth.

LEONARD WALDO.
FRANK A. GOOCH.

Witnesses to signature of Leonard Waldo:
FRANK L. CRAWFORD,
MARION FOWLER.
Witnesses to signature of Frank A. Gooch:
SARAH W. GOOCH,
INA B. MORRISON.