

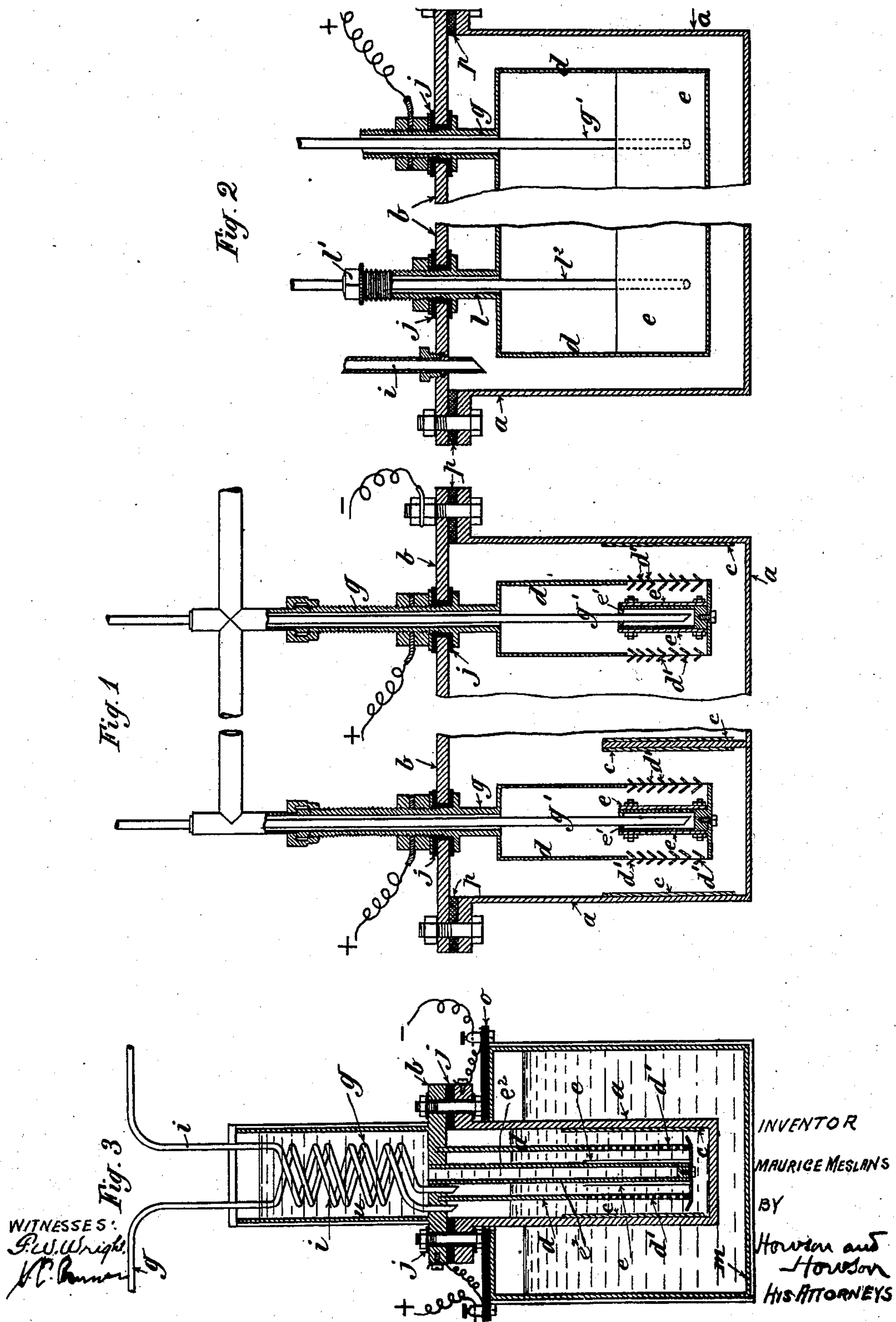
No. 692,688.

Patented Feb. 4, 1902.

M. MESLANS.
APPARATUS FOR PRODUCING FLUORIN.

(Application filed Nov. 10, 1900.)

(No Model.)



UNITED STATES PATENT OFFICE.

MAURICE MESLANS, OF PARIS, FRANCE.

APPARATUS FOR PRODUCING FLUORIN.

SPECIFICATION forming part of Letters Patent No. 692,688, dated February 4, 1902.

Application filed November 10, 1900. Serial No. 36,093. (No model.)

To all whom it may concern:

Be it known that I, MAURICE MESLANS, a citizen of the Republic of France, and a resident of Paris, France, have invented an Improved Apparatus for Producing Fluorin, of which the following is a full, clear, and exact description.

Fluorin was isolated for the first time in 1886 by Moissan. Hitherto this gas has been prepared by electrolyzing in a U-shaped tube a mixture of hydrofluoric acid and fluorid of potassium. This preparation constitutes a laboratory operation.

The present invention relates to an apparatus which enables fluorin to be made on an industrial scale.

The manufacture of fluorin presents three serious difficulties, which must be overcome. These difficulties consist, first, in the action of hydrofluoric acid and fluorin on the joints intended to insure the tightness of the apparatus and on the insulating washers or fittings in the case of electrical apparatus; second, in the phenomenon of counter-electrolysis due to the metallic partitions or walls of the apparatus, and, third, in the great resistance of the layer of electrolyte in a U-shaped tube.

As regards the first difficulty, the action of hydrofluoric acid necessitates the exclusion of insulating mineral products, with the exception of fluor-spar, which itself disintegrates rapidly, while the action of fluorin necessitates the exclusion of products having an inorganic base, such as sulfur and the like. In order to overcome this difficulty, I have constructed my apparatus with a view to dispensing with the presence of insulating parts and joints in the anode-cell, where the fluorin is formed. My invention enables this gas to be conveyed out of the apparatus, only allowing it to encounter metallic walls, on which it has no action or only a temporary action, the substance formed protecting the real casing, as will be hereinafter explained. For the joints of the cathode-cell, where the vapors of hydrofluoric acid are formed, I utilize materials having as a base organic products.

With the object of avoiding the effects of counter-electrolysis, which diminish the yield of fluorin, I employ for separating the anode and cathode spaces, and consequently the hydrogen gas and fluorin, a box having walls of

copper containing the anode. This cell or box is connected with the positive pole, and its walls are coated from the commencement of the electrolysis with a layer of fluorid of copper, forming a bad conducting covering, which enables them to act in an absolutely indifferent manner.

Naturally any other suitable metal having the property which has just been mentioned might be substituted for copper in the manufacture of the box in question. I construct the parts which are situated above the level of the electrolyte as actual or solid diaphragms, thus preventing the hydrogen gas and the fluorin from mixing.

The apparatus which I have designed permits of a reduction of the electrolytic resistance by the increase of the active surfaces of the electrodes and by the diminution of the thickness of the layer of electrolyte which the current has to traverse.

In the accompanying drawings, Figures 1 and 2 are vertical, longitudinal, and transverse sections, respectively, of an apparatus for production on a large scale. Fig. 3 also shows a vertical section of an apparatus of the same kind for production on a smaller scale.

In Figs. 1 and 2, *a* is a metallic vessel constituting the cathode-cell and containing the electrolyte, a mixture of anhydrous hydrofluoric acid and a metallic fluorid. It is closed by a lid *b* with a joint *p* of materials having organic products as a base. I prefer to employ caoutchouc for the insulating-joints. For the electrolyte I prefer anhydrous hydrofluoric acid containing about twenty-five per cent. of fluorhydrate or fluorid of potassium. In the receptacle *a* a suitable number of anode-cells *d* are arranged, placed parallel to one another, and the cathode-space contained between the two receptacles *d* is divided into two parts by intermediate walls or partitions. *c* indicates the cathodes, and *e* the anodes. The cells *d* are supported by two tubes *g* and *l* in communication with the anode-cell and connected with the positive pole. They are mounted on the lid *b* and electrically insulated from this latter by washers *j* of material having a base of organic substance forming a tight joint. The tube *g* serves for the discharge of the fluorin formed, and the other, *l*,

which is usually closed by a screw-stopper *l'*, serves when the generation is stopped for expelling the fluorin before opening the apparatus. A third tube *i*, also mounted on the lid *b*, communicates with the cathode-cell and serves for discharging the hydrogen. The cathodes *c* are connected with the negative pole by the walls of the receptacle *a*, and the current is brought to the anodes *e* by the pipes *g* and *l* and by the walls of the anode-cell, which are of suitably-selected metal, as is hereinbefore specified. The cells *d* above the level of the electrolyte are closed boxes, except at the outlets *g*, while below the level of the electrolyte the side walls of the cells consist of thin strips *d'* of a V-section arranged one beneath the other, the opening turned upward and fixed on the end walls. This arrangement assists in maintaining the separation of the gases and offers little resistance to the passage of the current.

With the object of cooling the electrodes the whole apparatus may be immersed in a refrigerating-tank, and a current of refrigerating liquid may be created in the hollow boxes *e'*, carrying the anodes, by means of pipes *g'* and *l'*, arranged within the pipes *g* and *l* and communicating with an external reservoir. These pipes, which are of the same metal as the anode-cell, also serve to convey electric current to the anode *e*.

In Fig. 3, which shows the arrangement for production on a small scale, the same letters are employed for indicating the like parts as in Figs. 1 and 2. This apparatus comprises only a single anode-cell, the wall of which beneath the level of the electrolyte is simply provided at *d'* with perforations of any suitable dimensions; but above the level of the electrolyte the cell is closed, except at the pipe *g*. The apparatus is immersed in a refrigerating-tank *m* and is supported by an ebonite plate *o*. It is surmounted by a refrig-

erator *u*, in which the pipes *g* and *i* coil spirally before emerging from the apparatus. The object of this arrangement is to avoid the carrying off of hydrofluoric acid by condensing it, so that it falls back in liquid form into the apparatus. This arrangement of a condensing-refrigerator is applicable to the different forms of the apparatus whether large or small.

The small apparatus of course does not possess a pipe for expelling the fluorin after each operation.

To cool the anode, this latter is constructed as a ring fitted on a tube *e'*, firmly attached to the cover *b* and in which the liquid contained in the refrigerator *u* can circulate.

I claim as my invention—

1. An apparatus for the manufacture of fluorin by electrolysis and provided with a partition wholly of metal separating the anode and cathode cells, and having openings through it only below the normal level of the electrolyte, said partition being electrically connected with the anode and being of a metal having the described property of forming on its surface a layer of insulating fluorid, all substantially as set forth.

2. An apparatus for the manufacture of fluorin by electrolysis and provided with a copper partition separating the anode and cathode cells, said partition being electrically connected with the anode and having openings below the normal level of the electrolyte but being imperforate above, all as and for the purpose described.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

MAURICE MESLANS.

Witnesses:

JOSEPH DELAGE,
EDWARD P. MACLEAN.