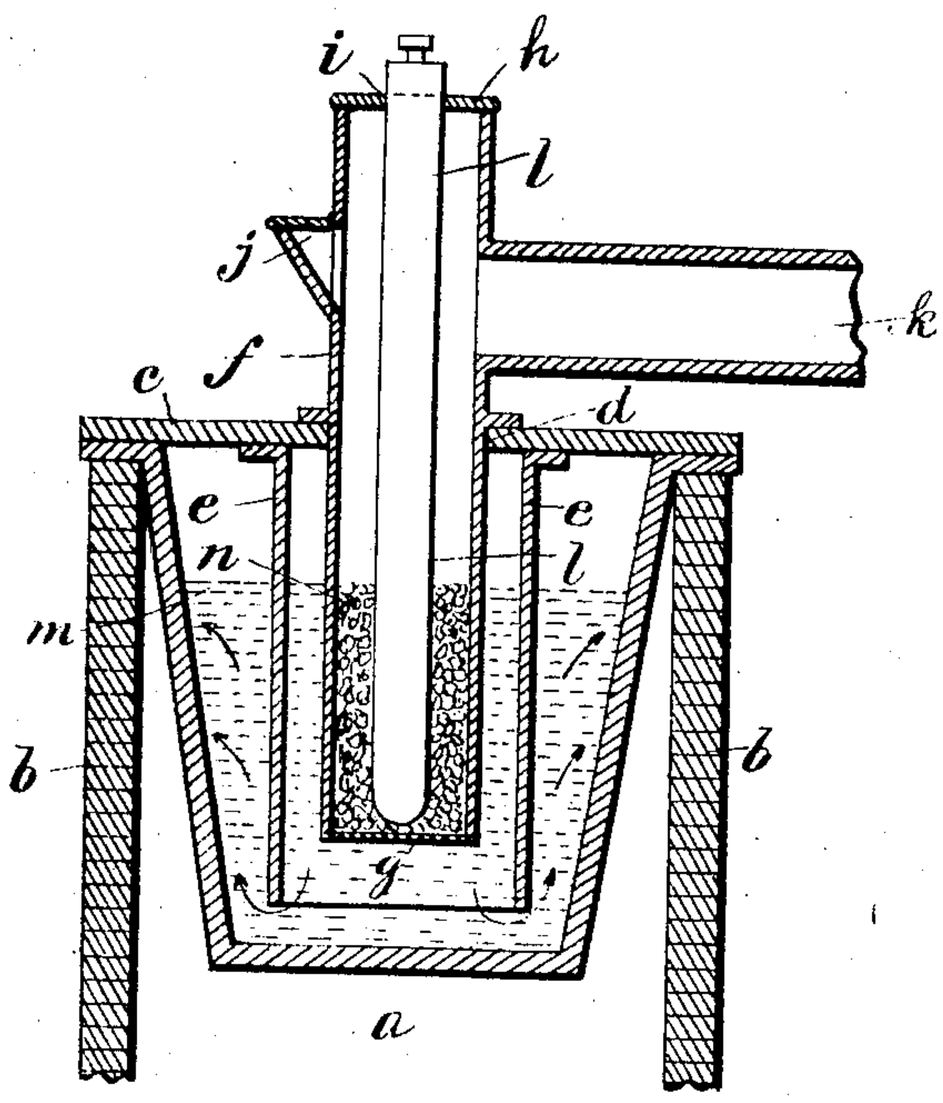
No. 785,961. PATENTED MAR. 28, 1905.

J. A. LYONS & E. C. BROADWELL.

MANUFACTURE OF CARBON TETRAFLUORID GAS.

APPLICATION FILED SEPT. 16, 1903.



Witnesses

Jas. A. Blackwood

Walter A. Blackwood.

Inventors

John A. Lyons

Edward C. Broadwell

By W. W. Dodge & Son

Attorneys

No. 785,961. Patented March 28, 1905.

UNITED STATES PATENT OFFICE.

JOHN A. LYONS AND EDWARD C. BROADWELL, OF CHICAGO, ILLINOIS.

MANUFACTURE OF CARBON-TETRAFLUORID GAS.

SPECIFICATION forming part of Letters Patent No. 785,961, dated March 28, 1905.

Application filed September 16, 1903. Serial No. 173,386.

To all whom it may concern:

Be it known that we, JOHN A. LYONS and EDWARD C. BROADWELL, citizens of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented new and useful Improvements in Manufacture of Carbon-Tetrafluorid Gas, of which the following is a specification.

Our invention relates to the process of producing, by means of electrolysis, carbon-tetrafluorid gas from the fluorids of the highly-electropositive alkaline, alkaline earth, and earth metals; potassium, sodium, lithium, calcium, barium, strontium, magnesium, manganese, chromium, and aluminium being produced at the same time as by-products.

While we are aware the fluorids have been used heretofore as a solvent for the oxids of the metals to be obtained electrolytically and that fluorids without such oxid or other compounds dissolved in them have been decomposed by an electric current, no attempt, to our knowledge, has been made to combine the fluorin gas with carbon to obtain the very valuable carbon-tetrafluorid gas.

The object of this present invention is to so combine fluorin gas and carbon that carbon-tetrafluorid gas may be produced at a low cost and rendered commercially available for the cheap production of artificial alcohol, acetic acid, formaldehyde, &c.

For a full description of the invention and the merits thereof and also to acquire a knowledge of the means for effecting the result reference is had to the following description, and to the accompanying drawing, in which a sectional view of our preferred form of electrolytic cell is shown; but we do not wish to limit ourselves to any particular form of cell or material.

Referring to the drawing, *a* is a crucible formed of any suitable metal in common use adapted for the purpose, which rests upon a wall *b*, within which may be built a fire (not shown) for heating the crucible and keeping its contents in a molten condition. Upon the crucible rests a cover *c*, of earthenware or other non-conducting material, provided with a central opening *d* about which, on the under side of the cover, is attached a partition *e*, of graphite or other suitable material, not necessarily porous, which sets down within the crucible and reaches almost to the bottom thereof, as shown. Through the opening *d* in the cover *c* is slipped a vessel *f*, formed of a suitable non-conducting material, having an open-work bottom *g*, a cover *h*, provided with a central opening *i*, and having an opening *j* above the crucible *a* on one side, through which any substance desired may be fed, and a branch or exit *k*, also above the crucible *a*, for the escape of the gas to be formed. Through the opening *i* in the cover *h* is slipped a stick of carbon or graphite *l*, which reaches to the bottom of the vessel *f* and forms the anode of the electrolytic cell. We preferably use the inner lining of the crucible *a* for the cathode, although an extra rod or plate of any suitable material may be suspended therein, if so desired.

We prefer to make all the elements above described circular in section on account of cheapness of construction; but obviously they may be of any cross-section and our process be equally well conducted thereby.

In carrying out our invention a foundation-bath *m*, preferably of sodium and potassium fluorid, although the fluorids of any of the alkaline, alkaline earth, or earth metals may be used, and the fluorid CaF_4 is named as a suitable and practical fluorid to be used, is placed in the crucible *a* and kept in a molten state by means of any external heat and through the opening *j* is slipped a quantity *n* of charcoal, lampblack, or other suitable carbonaceous material, which surrounds the lower portion of the anode *l* and floats upon the surface of the bath *m*. The quantity of soft carbonaceous material added is sufficient to make, with the hard carbon or graphite, a perfect anode and to protect the latter from unnecessary disintegration. The electrodes are then connected in circuit to some suitable source of electrical energy, and the bath *m* is electrolyzed by a low-pressure current, about eight volts having been found sufficient. Thereupon the metal base—sodium, calcium, potassium, &c., according to the particular fluorid that may be used—will be deposited upon the cathode giving one or more of these

alkaline, alkaline earth, or earth metals as a by-product. At the same time fluorin gas being liberated at the anode instantly combines with the surrounding carbonaceous material, producing carbon-tetrafluorid gas, which may be led off through the exit or duct *k* to any suitable receiver. Since the combination of the fluorin with the carbon is instantaneous, there is practically no free fluorin gas in existence during the process. The reaction takes place at an approximate temperature of 1,000° centigrade.

It will be noted that surrounding the anode with the carbonaceous material *n* protects the comparatively costly anode from unnecessary waste, and thus cheapens the cost of production of the gas, besides performing its main function of furnishing carbon to the fluorin. Also the partition *c* effectually prevents loss arising from the fluorin or carbon-tetrafluorin gas reaching the deposited metals. The bath may be kept fused by any suitable means, the fire being shown as one form of such means, and the electric current may be made strong enough to maintain such state of fusion should it be desired.

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

1. The process of producing carbon-tetrafluorid gas from the fluorids of the electropositive metals, consisting in passing a current of electricity through a fused bath of the fluorid of such metal in the presence of a cathode and an anode and subjecting the fluorin gas liberated at the anode to the action of carbonaceous material immediately surrounding the anode.

2. The process of producing carbon-tetrafluorid gas from the fluorids of the electropositive metals, which consists in passing a current of electricity through the fluorid of any of said metals in the presence of a suitable cathode and a carbon anode and subjecting the fluorin gas liberated at the anode to a separate body of carbonaceous material immediately surrounding the anode, whereby carbon-tetrafluorid gas will be instantly produced and any molecular fluorin that may be created will be converted to said carbon-tetrafluorid gas, and the metals of the fluorids will be deposited at the cathode.

3. The process of producing carbon-tetrafluorid gas which consists in passing an electrolytic current through a fused bath of the fluorid of one of the electropositive metals and subjecting the fluorin gas liberated at the anode to a body of carbonaceous material having an affinity for fluorin.

4. The process of producing carbon-tetrafluorid gas which consists in passing an electrolytic current through a fused bath of a fluorid of an electropositive metal and subjecting the fluorin liberated at the anode to a mass of carbonaceous material having an affinity therefor, and immediately surrounding the anode.

5. The process of producing carbon-tetrafluorid gas which consists in passing an electrolytic current through metallic fluorid in a state of fusion and subjecting the fluorin liberated at the anode to a body of corbonaceous material having an affinity for fluorin immediately surrounding the anode.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

JOHN A. LYONS.
EDWARD C. BROADWELL.

Witnesses:
MATTHEW MURPHY,
LADIMIR MONDRY.