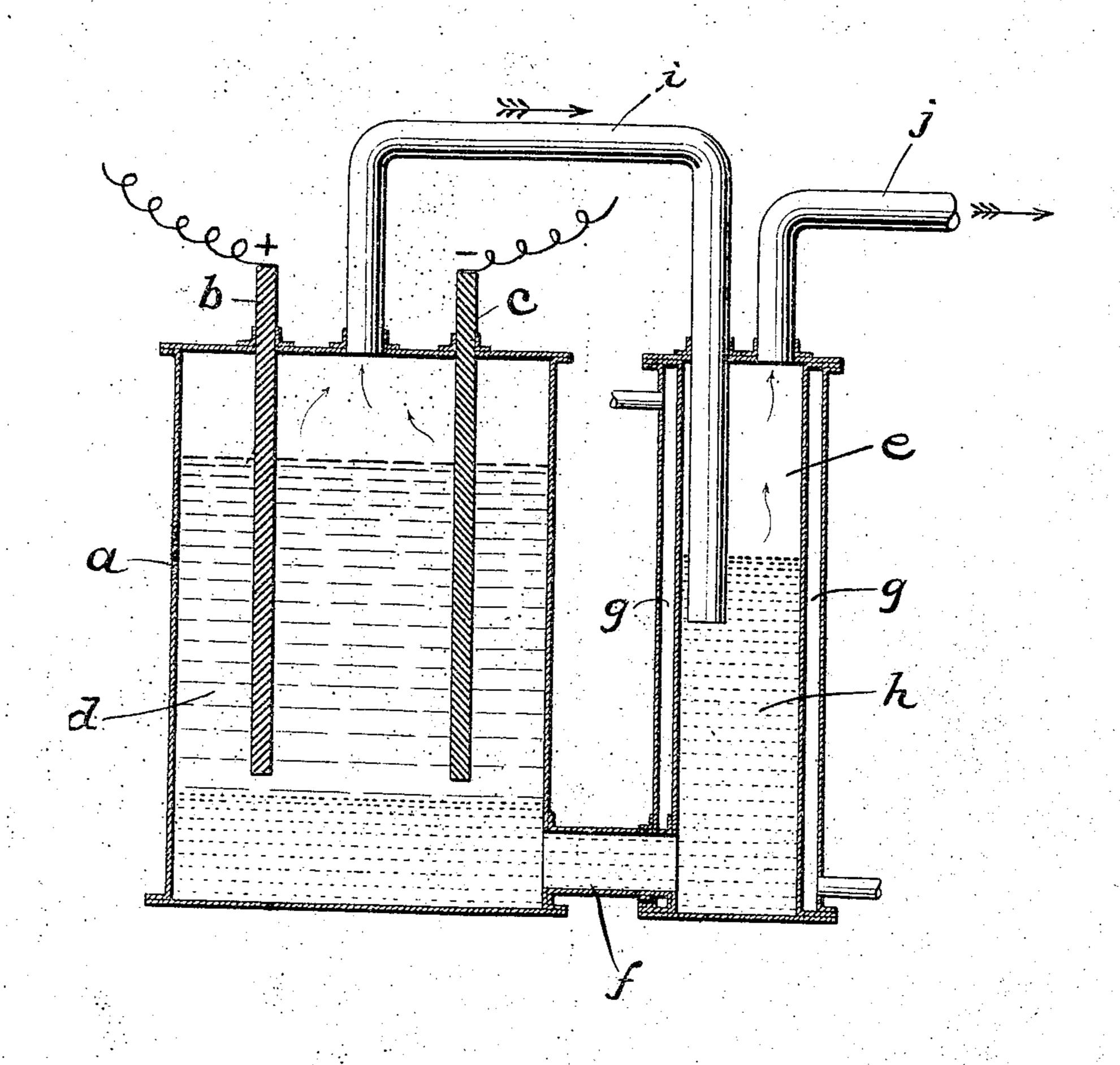
## M. PIER.

METHOD OF PRODUCING HALOGEN-OXYGEN COMPOUNDS BY ELECTROLYSIS.

APPLICATION FILED APR. 29, 1911.

998,982.

Patented July 25, 1911.



WITNESSES:

Rob Retichel

Mathias Piex

BY Freedy ATTORNEYS

## UNITED STATES PATENT OFFICE.

MATHIAS PIER, OF SCHLACHTENSEE, NEAR BERLIN, GERMANY, ASSIGNOR TO E. I. DU PONT DE NEMOURS POWDER COMPANY, OF WILMINGTON, DELAWARE, A CORPORA-TION OF NEW JERSEY.

METHOD OF PRODUCING HALOGEN-OXYGEN COMPOUNDS BY ELECTROLYSIS.

998,982.

Specification of Letters Patent. Patented July 25, 1911.

Application filed April 29, 1911. Serial No. 624,136.

To all whom it may concern:

Be it known that I, Mathias Pier, a subject of the Emperor of Germany, residing at Schlachtensee, near Berlin, Germany, 5 have invented a new and useful Improvement in Methods of Producing Halogen-Oxygen Compounds by Electrolysis, of which the following is a full, clear, and exact description, reference being had to the 10 accompanying drawings, which form a part

of this specification.

It is known that high yields of halogen oxygen compounds have been obtained by electrolysis by using precautions to prevent 15 the reaction of the hypo-halogen compounds at the cathode and thus the prevention of the formation of oxygen at the anode. It is also known that the reduction of the halogen compounds at the cathode is, to a great ex-20 tent, prevented by means of the addition of certain materials, for instance, chromate, to partly due to the fact that the temperature is too high, and partly to the fact that the 25 electrolyte, in most instances, does not react with neutral or very weak acid. No matter how the operation may be carried out, the maintenance of the electrolyte in this condition is always effected with great difficul-30 ties, due to the fact that in neutral, or in weak acid solution, chlorin escapes, until the solution reacts alkaline. It has been attempted to overcome this difficulty by passing a current of chlorin into or over the elec-35 trolyte, or from time to time adding hydrochloric acid. Both of these methods have the objection that the hypohalogen compounds are suddenly decomposed, evolving chlorin, and the reaction soon sets in again.

40 Further, constant watching is required. I overcome these difficulties and also maintain the electrolyte at a constant level by adding to the neutral electrolyte a liquid which is not miscible with the electrolyte but which 45 readily dissolves chlorin. This liquid should be previously saturated with chlorin. The gases developed, particularly chlorin, are brought into intimate contact with this liquid in such a manner that the gas does 50 not escape and is absorbed and returned. again to the electrolyte. The liquid or material used should be one which will absorb

chlorin gas and readily give it up. Tetra-

chlorid of carbon is a liquid which can be

used and is especially suited for this pur- 55 pose, but other liquids or solids which absorb chlorin may be added to electrolyte. If the operation is carried on at a temperature which will or may volatilize the added liquid, a reflux condenser should be used.

In practice I have used the following electrolyte or bath: neutral sodium chlorid solution diluted with 0.2% of chromate and 20 to 30 vol. per cent. of tetra chlorid of carbon. The anode is formed of platinum-iridi- 65 um and the cathode of graphite, all in a closed cell. With this I have obtained practically the theoretical yield of total halogenoxygen compound on current used, the chlorid content being about 1%.

In the drawing is represented in section an apparatus for carrying out my method.

a is the electrolyzing cell, b the anode, c the cathode, d the electrolyzing solution, e is a separate receptacle, having communica- 75 the electrolyte. The formation of oxygen is | tion with the interior of the electrolyzing cell, by means of passage f.

g are the jacketed walls surrounding the liquid h, tetra chlorid of carbon, in the receptacle e for cooling the liquid.

i is a pipe communicating with the upper portion of the electrolyzing cell a, and entering the upper portion of liquid h in receptacle e.

j is an outlet pipe for the passage of hy- 85 drogen gas.

The hydrogen and chlorin gases which form in the cell a pass out through the pipe i into the liquid in receptacle e. In this liquid the chlorin gas is absorbed, and by 90 reason of the diffusion, is led back to the electrolyte through the passage f.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:

1. The improvement in the method of producing halogen-oxygen compounds by electrolysis, which consists in adding to the electrolyte a substance which absorbs and readily releases chlorin gas.

2. The improvement in the method of producing halogen oxygen compounds by electrolysis, which consists in allowing the evolved chlorin gases to escape from the cell, absorbing the same in a substance which ab- 105 sorbs and readily gives up said chlorin gas, and returning the gas to the electrolyte. 3. The improvement in the method of

producing halogen-oxygen compounds by electrolysis, which consists in adding to the electrolyte a liquid which absorbs and

readily releases chlorin gas.

4. The improvement in the method of producing halogen oxygen compounds by electrolysis, which consists in allowing the evolved chlorin gases to escape from the cell, absorbing the same in a liquid which absorbs and readily gives up said chlorin gas, and returning the gas to the electrolyte.

5. The improvement in the method of producing halogen-oxygen compounds by electrolysis, which consists in adding to the electrolyte a liquid which is not miscible with the electrolyte, and which absorbs and

readily releases chlorin gas.

ducing halogen oxygen compounds by electrolysis; which consists in allowing the evolved chlorin gases to escape from the cell, absorbing the same in a liquid, which is not miscible with the electrolyte, and which absorbs and readily gives up said chlorin gas, and returning the gas to the electrolyte.

7. The improvement in the method of producing halogen oxygen compounds by electrolysis, which consists in adding to the elec-

trolyte tetra chlorid of carbon.

30 8. The improvement in the method of pro-

ducing halogen oxygen compounds by electrolysis, which consists in allowing the evolved chlorin gases to escape from the cell, absorbing the same in tetra chlorid of carbon, and returning the gas to the electrolyte. 35

9. The improvement in the method of producing halogen-oxygen compounds, which consists in providing a liquid independent of the electrolyte, and which liquid absorbs and readily gives up chlorin, said liquid being in 40 liquid connection with the lower portion of the electrolyte and in gas communication with the upper portion of the electrolyzing cell.

10. The improvement in the method of 45 producing halogen-oxygen compounds, which consists in providing tetra chlorid of carbon independent of the electrolyte, said tetra chlorid of carbon being in liquid connection with the lower portion of the electrolyte 50 and in gas communication with the upper portion of the electrolyzing cell.

In testimony of which invention, I have hereunto set my hand, at Berlin, on this 6

day of April, 1911.

MATHIAS PIER.

Witnesses:

WOLDEMAR HAUPT, HENRY HASPER.