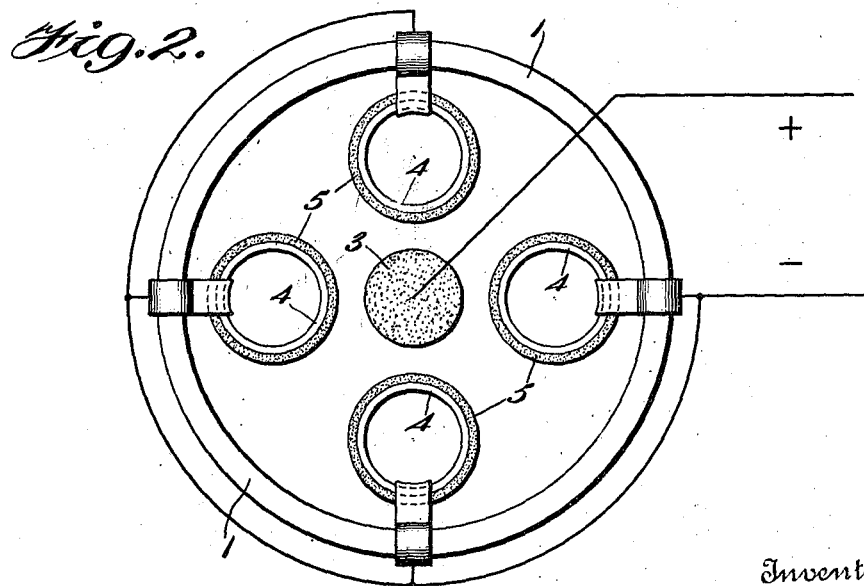
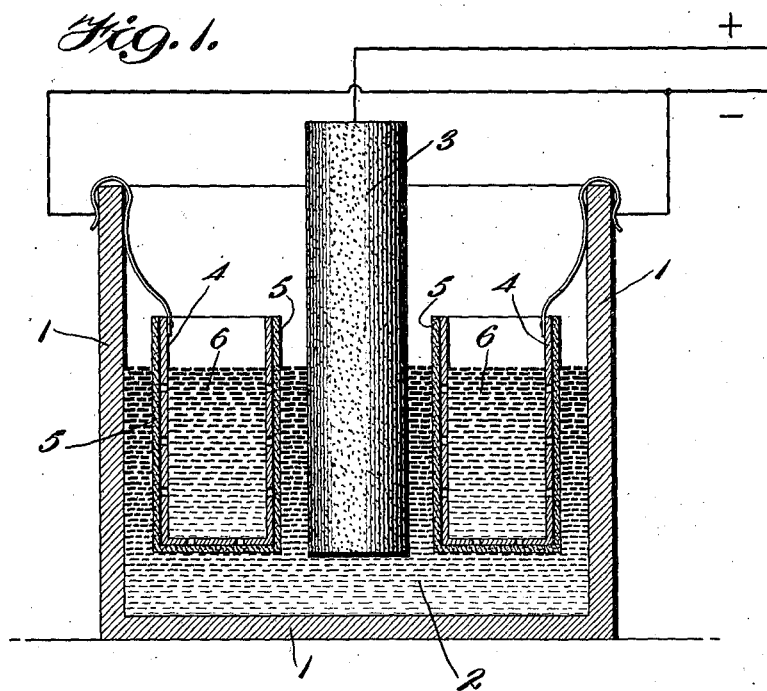


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 PROCESS OF PRODUCING PERHALATES.
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To all whom it may concern:

Be it known that we, EUGENE PAUL SCHOCH and RUFUS HUBBARD PRITCHETT, both citizens of the United States, both residing at Austin, in the county of Travis and State of Texas, have invented certain new and useful Improvements in Processes of Producing Perhalates; 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 invention relates to a process of producing perhalates, and especially perchlorates, and their corresponding acids, through the use of an anode composed essentially of carbon, or silicon, and has for its object to provide a method which will be simple to carry out and more economical in practice than those heretofore proposed.

With these and other objects in view the invention consists in the novel steps and combinations of steps involving the use of a carbon or silicon anode, and constituting the process, all as will be more fully hereinafter disclosed and particularly pointed out in the claims.

Referring to the accompanying drawings forming a part of this specification in which like numerals designate like parts in all the views:—

Figure 1 is a diagrammatic sectional view of an apparatus suitable for carrying out the invention; and—

Fig. 2 is a plan view of the parts shown in Fig. 1.

1 indicates any suitable container, 2 any suitable electrolyte, such for example, as sodium chlorate, NaClO_3 , which may have an impurity such as sodium chlorid mixed therewith, 3 any suitable carbon or silicon, or chemically similar anode, 4 any suitable metallic cathode such as copper gauze, nickel, iron, etc., and 5 a porous cup or other diaphragm surrounding the cathode 4, to separate the anode liquid from the cathode.

In order that the precise invention may be the more clearly understood it is said:— Prior to our invention, it has been erroneously believed that no commercial quantity of perchlorates could be produced in an electrolytic cell using a carbon anode. This erroneous belief has been founded partly on the fact that the oxygen liberated at the carbon anode in an acid solution gives rise to a disintegration, or oxidation, of the carbon

material; and hence it was thought that such disintegration would be sufficient to destroy the anode to such an extent as to make the process impracticable commercially.

Winteler, in *Zeitschrift für Elektrochemie*, vol. 7 of 1901, p. 635, is authority for, and is probably one of the chief sources of the belief that a carbon anode cannot be substituted for platinum anode in an acid electrolyzing solution when producing perchlorates on a commercial scale.

Further, it is a well recognized fact, that, if the solution is maintained neutral or alkaline throughout the process, the discharge potential from a carbon anode will not be great enough to commercially form perchlorates. Winteler really recognizes this fact also, for he states broadly that perchlorates cannot be formed at all with carbon electrodes.

We have discovered, on the other hand, that it is perfectly feasible to substitute carbon, or silicon, for platinum as anodes, in the commercial production of perchlorates, and that therefore the present very high cost that the use of platinum entails may be avoided, not only in the commercial production of perchlorates in general, but also in the commercial production of perhalates.

In carrying out our process, we employ an anode consisting essentially of carbon, or of silicon, and prefer to start with an acid solution of the chlorate corresponding to the perhalate to be made, but of course, we may start with a neutral, or even an alkaline solution, and permit the action of the current to render the solution acid as the process proceeds.

Referring to the drawings, we prefer to employ a current strength of from say three to twenty amperes per one hundred square centimeters on the anode surface, and it is also preferred to keep the solution cold during the entire process. As the current passes the usual cathode reactions take place in the hydroxid solution 6, provided of course, such a solution surrounds the cathodes 4; and at the anode 3 likewise, the usual reactions will take place which in this case consists of the discharge of the oxygen ions from the water, and the reaction of this oxygen with the chlorate ions at the surface of the carbon anode. This last mentioned reaction will form perchlorate ions and free electrons which pass out through the pole. The hydrogen ions left from the water molecules

of which the oxygen has been used up, remain free in the solution and render the same acid in character.

We have found by actual tests that with an apparatus constructed as above described, provided the anode is surrounded by any appreciable quantity of the acid solution, we can, on a commercial scale, continue to convert the chlorate until practically the whole of it is changed into perchlorate, for our tests show a conversion efficiency of say 50% or greater of the total amount of current employed. On the other hand these results are impossible if the anode is in contact with an alkaline solution.

It will thus be seen that contrary to the statement of Winteler as well as of other authorities, not necessary to mention, and certainly contrary to the common belief among those skilled in the art, the substitution of carbon or silicon for the now almost prohibitive platinum is perfectly feasible in the manufacture of perchlorates at a commercial efficiency.

Our tests further show that although a small decomposition of the carbon anode actually takes place, yet, it has not proved to be at all prohibitive, and in some cases it has been as low as 9% by weight of the weight of the perchlorate produced.

In the absence of porous diaphragms, such as 5, the alkali produced by the pole reactions at the cathode will, of course, tend to neutralize the acid produced at the anode, but the acidification of the liquid around the anode will increase unless the two liquids are properly mixed. If said liquids are mixed, of course, substantially no perchlorate at all will be formed. But, if the liquids are not mixed, the acidification around the anode will increase as the process continues, and will depend upon the distance the anode and cathode are separated.

In other words, when the liquids are not mixed, a slight acidity may be maintained around the anode and this acidity may be increased by the addition of acid from an extraneous source. Under these conditions, perchlorate has also been obtained by us in appreciable quantities. But, in such acidified solutions, the chlorate that comes in contact with the cathode is reduced to the chlorid, and when the chlorid thus formed comes in contact with the anode again, it becomes oxidized and thus it entails a waste of current in so far as the formation of perchlorate from chlorate is concerned.

Furthermore, this formation of the chlorid is objectionable for we have found in carrying out our process that the extensive simultaneous oxidation of the chlorids at the anode increases appreciably the carbon anode decomposition. Hence to overcome both of these sources of trouble, we have separated the cathodes 4 by porous diaphragms 5 from

the anode liquid, thus preventing access of the acidified chlorate solution to the cathode and its consequent reduction to the chlorid.

It will now be clear that by operating as above disclosed we are enabled to substitute the non-metallic elements of the carbon group, *e. g.*, carbon, and silicon, for the expensive platinum heretofore thought necessary for the production of perchlorates, and therefore, we are enabled to commercially produce not only a perchlorate of sodium or potassium at a much less cost in the plant than was heretofore thought to be possible, but we are enabled to produce perhalates in general.

We have further discovered that the decomposition of the carbon and silicon anodes may be greatly diminished and the current efficiency increased, by the following treatment, preliminary to their use:—heating the anodes, plunging them into a bath of melted paraffin, ceresin, or ozocerite, and allowing them to remain until cool, whereupon the outer conducting surface may be exposed by mechanically cleaning off the excess of wax-like material.

This treatment not only prevents the solution from penetrating into the pores of the anodes, thus causing a mechanical disintegration by the evolution of gases therein, etc., but it also limits the active surface to an outer surface of known extent, and makes it possible to provide a definite current density. This latter is very desirable, because, too low or too high a current density entails a lower current efficiency.

We have found the most efficient form of carbon for our process to be that known as retort carbon. In fact, the retort carbon has been found to possess the surprising quality of being a plurality of times more efficient for this particular purpose than Acheson graphite. Of course, it is obvious that periodates, perbromates, and their corresponding acids may be produced in the same manner as the perchlorates, and the perchloric acid above mentioned; and it is further obvious that in addition to the employment of alkali metal chlorates as electrolytes, we may use any soluble chlorates, including chloric acid.

It is obvious that those skilled in the art may vary the process without departing from the spirit thereof, and therefore, we do not wish to be limited to the above disclosure except as may be required by the claims.

What we claim is:—

1. The process of producing a perhalate in commercial quantities which consists in electrolyzing the corresponding halate solution with an anode comprising a non-metallic element of the carbon group and in contact with an acid solution, substantially as described.

2. The process of producing a perhalate in a commercial quantity which consists in electrolyzing the corresponding halate solution with an anode containing carbon and
5 in contact with an acid solution, substantially as described.

3. The process of producing a perchlorate which consists in electrolyzing the corresponding acid chlorate solution with an
10 anode comprising carbon, substantially as described.

4. The process of producing a perchlorate of an alkali metal which consists in electrolyzing the corresponding acid chlorate solution with a carbon anode, substantially as
15 described.

5. The process of producing sodium perchlorate which consists in electrolyzing an acid solution of sodium chlorate with an anode of gas retort carbon, substantially as
20 described.

6. The process of producing sodium perchlorate and perchloric acid which consists in electrolyzing an acid solution of sodium chlorate, with an anode of carbon, and separating the anode liquid from the cathode,
25 substantially as described.

In testimony whereof we affix our signatures.

EUGENE PAUL SCHOCH.
RUFUS HUBBARD PRITCHETT.