

# UNITED STATES PATENT OFFICE.

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## PROCESS OF ELECTROLYTICALLY PRODUCING CHROMIC ACID FROM CHROMIC SULFATE.

No. 895,930.

Specification of Letters Patent.

Patented Aug. 11, 1908.

Application filed April 27, 1907. Serial No. 370,739.

*To all whom it may concern:*

Be it known that we, GUSTAV ADOLPH, of Ammendorf, doctor of philosophy, a subject of the King of Prussia, and whose post-office address is Schachtweg, Ammendorf, near Halle, Prussia, German Empire, and ALBERT PIETZSCH, of Magdeburg, a subject of the King of Saxony, and whose post-office address is No. 34 Kaiser-Otto-Ring, Magdeburg, Prussia, German Empire, have invented a new and useful Process of Electrolytically Producing Chromic Acid from Chromic Sulfate; and we do hereby declare that the following is a full, clear, and exact description of our invention, which will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to the process of electrolytically producing chromic acid from chromic sulfate, and it has for its object to so improve this process that it can be carried out with an acid condition of the electrolyte and in the absence of a diaphragm between the electrodes.

Our improved process is especially intended for recovering chromic acid from the liquors containing chromic sulfate which are obtained in the well-known process of oxidizing organic compounds by means of chromic acid. According to the electrolytical process actually in use for this purpose the chromic sulfate solution is electrolyzed by means of lead electrodes in a lead vessel subdivided by a diaphragm in an anode chamber and a cathode chamber, the electrolyte being maintained in a strongly acid condition throughout the process to give it a good electrical conducting power. By the passage of the electric current through the said solution the chromic sulfate is split up into chromic oxid and sulfuric acid and the chromic oxid is oxidized to chromic acid at the anode, the sulfuric acid set free accumulating in the liquor at the anode. When the oxidizing process is finished, the liquor contained in the anode chamber is withdrawn therefrom to the outside, whereas the liquor contained in the cathode chamber is transposed into the anode chamber, and the cathode chamber is supplied with fresh chromic sulfate solution. This process is defective in that the diaphragm augments the inner electrical resistance and, moreover, is subject to a rapid

wear, so that it must be renewed after some use.

It is well known to electrolytical experts that up to the present invention all attempts have failed to obtain a satisfactory result by electrolyzing chromic sulfate solutions in an acid condition without the presence of a diaphragm for the reason that no means other than the interposition of a diaphragm between the electrodes were available for protecting the chromic acid formed against the reducing action of the hydrogen set free. Now we have discovered that such protection will be obtained in a very efficient manner by the presence in the chromic sulfate solution of certain alkaline salts, namely alkaline sulfates or alkaline acetates and mixtures of both. This effect of the presence of said alkaline compounds in the chromic sulfate solution is due on the one hand, to a reduction of the cathodic potential, it being well known that the reducing capacity of the nascent hydrogen increases and diminishes according as the cathodic potential is higher or lower; on the other hand, alkaline bichromate is formed as the main oxidation product and this opposes a greater resistance to the reducing action of the nascent hydrogen than chromic acid, and, moreover, does not react with free acetic acid and dilute sulfuric acid in the absence of heat.

Therefore our present invention essentially consists in electrolyzing chromic sulfate solutions in the absence of a diaphragm between the electrode but in the presence of alkaline acetates, or alkaline sulfates or mixtures of both, the liquor being maintained in its acid condition throughout the process.

Besides dispensing with the use of a diaphragm and thus eliminating the many inconveniences connected therewith, our present invention has the following advantages: The apparatus is simplified and less costly; the liquor is continuously agitated by the escaping hydrogen whereby the anode is continuously supplied with fresh liquor and consequently a very perfect utilization of the nascent oxygen is secured, and finally the electrodes may be arranged at a proximity to each other whereby in connection with the absence of the diaphragm a low current tension is required for carrying out the process.

In general we prefer to use mixtures of

alkaline acetates with alkaline sulfates, especially sodium sulfate as this is cheap and, moreover, has the effect of notably augmenting the electrical conducting-power of the chromic sulfate solution.

We proceed to illustrate the efficiency of our improved process by some examples:

1. The electrolyte is composed so as to contain 400 grams per liter chromic sulfate ( $\text{Cr}_2(\text{SO}_4)_3 + \text{aq.}$ ), 150 grams per liter sodium sulfate ( $\text{Na}_2\text{SO}_4 + \text{aq.}$ ), 150 grams per liter sodium acetate ( $\text{NaC}_2\text{H}_3\text{O}_2 + \text{aq.}$ ) This liquor is electrolyzed by means of lead electrodes, applying a current density of 200 amperes per square meter at the anode, and progressively increasing the current density at the cathode from 200 amperes per square meter at the beginning up to 2000 amperes per square meter according as the per cent. of chromic acid increases in the electrolyte. In this way about 100 grams per liter of chromic acid will be obtained and 90 per cent. of the electric current utilized.

2. The electrolyte is composed so as to contain 500 grams per liter chromic sulfate—( $\text{Cr}_2(\text{SO}_4)_3 + \text{aq.}$ ), 200 grams per liter ammonium sulfate—( $\text{NH}_4)_2\text{SO}_4$ , 100 grams per liter sodium acetate  $\text{NaC}_2\text{H}_3\text{O}_2 + \text{aq.}$  Under like conditions as in the first example 110 grams per liter of chromic acid will be obtained and 85 per cent of the electric current utilized. With an electrolyte of a smaller percentage of chromic acid still better results will be obtained.

What we claim as our invention is:

1. The process of electrolytically producing chromic acid from chromic sulfate which comprises mixing with a chromic sulfate solution an alkaline salt capable of reducing cathodic potential and electrolyzing said mixture in the absence of a diaphragm, whereby the reducing action of the nascent hydrogen is minimized and the electrolyte maintained acid during the process.

2. The process of electrolytically producing chromic acid from chromic sulfate which consists in adding to the chromic sulfate solution a mixture of alkaline salts capable of reducing cathodic potential and electrolyzing

said mixture in the absence of a diaphragm, whereby the reducing action of the nascent hydrogen is minimized and the electrolyte maintained acid during the process.

3. The process of electrolytically producing chromic acid from chromic sulfate which consists in adding to the chromic sulfate solution a mixture of an alkaline sulfate and an alkaline acetate capable of reducing cathodic potential and electrolyzing said mixture in the absence of a diaphragm, whereby the reducing action of the nascent hydrogen is minimized and the electrolyte maintained acid during the process.

4. The process of electrolytically producing chromic acid from chromic sulfate which consists in adding to the chromic sulfate solution a mixture of sodium sulfate and sodium acetate and electrolyzing said mixture in the absence of a diaphragm, whereby the reducing action of the nascent hydrogen is minimized and the electrolyte maintained acid during the process.

5. The process of electrolytically producing chromic acid from chromic sulfate which comprises mixing with a chromic sulfate solution sodium sulfate and electrolyzing said mixture in the absence of a diaphragm, whereby the reducing action of the nascent hydrogen is minimized and the electrolyte maintained acid during the process.

6. The process of electrolytically producing chromic acid from chromic sulfate which comprises electrolyzing a chromic sulfate solution in the absence of a diaphragm and in the presence of alkaline salt having the property of reducing the cathodic potential, protecting the chromic acid formed from the reducing action of the nascent hydrogen and maintaining the electrolyte acid during the process.

In testimony whereof we have hereunto set our hands in presence of two subscribing witnesses, viz., for both the inventors.

GUSTAV ADOLPH.  
ALBERT PIETZSCH.

Witnesses:

CARL LINDEMANN,  
GUSTAV ESCHÉ.