JULIUS TAFEL, OF WÜRZBURG, GERMANY, ASSIGNOR TO C. F. BOEHRING & SOHNE, OF MANNHEIM-WALDHOF, GERMANY, A FIRM.

ART OF PRODUCING HYDROXYLAMIN.


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To all whom it may concern:

Be it known that I, JULIUS TAFEL, a citizen of the German Empire, residing at Würzburg, Bavaria, Germany, have invented certain new and useful Improvements in the Art of Producing Hydroxylamin; and I 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 pertains to make and use the same.

My invention relates to the art of producing hydroxylamin.

The object of my invention is to produce hydroxylamin by electrolytic reduction.

It has not been possible hitherto to obtain hydroxylamin as a reduction product by the electrolytic reduction of free nitric acid.

I have discovered now that hydroxylamin may be obtained in good quantities by electrolysis under suitable conditions of a solution containing an acid electrolyte in addition to the nitric acid to be reduced. By my process, therefore, it is possible to produce directly the said hydroxylamin on a commercial scale and from nitric acid.

In practicing my invention it is necessary to carry out the reduction while well cooling. Furthermore, it is necessary to employ care that the electrolyte be quickly and uniformly stirred and that the possibilities of an excess of nitric acid be avoided. In order to comply with the latter requirement, the nitric acid is added slowly in the proportion which is to be used, so that it is supplied only at one concentration just sufficient for the most advantageous employment of the current. As cathode material I have found either mercury or an amalgamated cathode as specially suitable, although the reduction may also be carried out by the employment of a cathode of lead, tin, silver, nickel, bismuth, or copper. Other by-products of the reduction of nitric acid are produced by my process in more or less subordinate quantities, according to the material of the cathode used.

In order to enable these skilled in the art to fully understand my invention, I will now give a detailed description of the same in connection with two examples embodying what I now consider to be the preferred method of carrying the same into effect.

I. Reduction of nitric acid to hydroxylamin in sulfuric-acid solution.—An electrolytic cell is separated into an anode-chamber and a cathode-chamber by a diaphragm. The cathode-chamber is filled with six kilograms of fifty-per-cent. sulfuric acid, the anode-space with the same fifty-per-cent. sulfuric acid. Each chamber of the cell is provided with means for cooling. The cathode consists of a well-amalgamated lead, while the anode is formed of pure lead. A current of sixty to one hundred and twenty amperes per square decimeter is now sent through the bath, both spaces of the cell being well cooled. Thereupon two kilograms of a fifty-per-cent. solution of nitric acid are added slowly to the bath by a dropping apparatus, so that always a slight evolution of hydrogen is just noticeable at the cathode. At the same time the cathode solution is powerfully stirred. During the operation it is advisable to keep the temperature of the bath at about 20° centigrade. The reduction is carried out until a test of the cathode liquid proves there is no further nitric acid in it. The resulting hydroxylamin is then isolated from the reaction liquid as sulfate or chlorhydrate by known methods.

Example II.—Reduction of nitric acid to hydroxylamin in hydrochloric-acid solution.—In carrying out this example the same steps are employed as in Example I, with the exception, however, that the cathode-chamber is provided with six kilograms of about twenty-per-cent. solution of hydrochloric acid. It is preferable to employ a tin cathode or equivalent cathode for this example, or if a cathode of indifferent material is used then some spongy tin should be added to the bath in the proportion of about ten grams per liter.

Having thus fully described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of producing hydroxylamin, which consists in reducing nitric acid by electrolytic action.

2. The process of producing hydroxylamin, which consists in subjecting nitric acid to electrolytic action in the presence of another electrolyte.

3. The process of producing hydroxylamin, which consists in subjecting nitric acid to
4. The process of producing hydroxylamin, which consists in subjecting nitric acid to electrolytic action and cooling the bath.

5. The process of producing hydroxylamin, which consists in slowly adding nitric acid to an acid electrolytic bath while agitating and cooling said bath, and passing an electric current through the same.

6. The process of producing hydroxylamin, which consists in slowly adding nitric acid to a solution of hydrochloric acid in a cathode-chamber, while stirring and cooling, and passing an electric current through the same.

7. The process of producing hydroxylamin, which consists in gradually adding nitric acid to an acid electrolytic bath while cooling, stirring and passing an electric current through the bath, the nitric acid being added in such quantities that a continual slight evolution of hydrogen occurs at the cathode.

In testimony whereof I affix my signature in presence of witnesses.

JULIUS TAFEL.

Witnesses:
L. Heller,
Katharina Schatz,
Jacob Adrian.