UNITED STATES PATENT OFFICE.

MAX BUCHNER, OF MANNHEIM, GERMANY.

REDUCTION OF NITRO COMPOUNDS.

SPECIFICATION forming part of Letters Patent No. 700,672, dated May 20, 1902.

Application filed September 24, 1900. Serial No. 30,968. (No specimens.)

To all whom it may concern:

Be it known that I, MAX BUCHNER, a citizen of the Empire of Germany, residing at Mannheim, in the Empire of Germany, have invented certain new and useful Improvements in the Reduction of Nitro Compounds; 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 appertains to make and use the same.

This invention relates to the reduction of nitro compounds, with particular reference to the employment of an electric current for such purpose.

In my application for Letters Patent of the United States, Serial No. 737,494, filed November 18, 1899, I have described a process of electrolytic reduction of nitro compounds which consists, essentially, in causing an electric current to pass through the nitro compound placed in the cathode-space of an electrolytic cell in the presence of tin, the tin being in acid solution. Under that process the reduction of the compound employed and the recovery of the tin employed take place simultaneously. In the course of my further experiments and researches in this direction I have found that this method may be carried out also by means of copper; and my present invention consists in the reduction of nitro compounds by causing the electric current to pass through such compounds in acid solution and in the presence of copper ions and in such further features as will be hereinafter set forth, and pointed out in the claims. Just as in the process set forth in my aforesaid application, No. 737,494, the cathode material is unimportant, since the same serves merely to convey the current, while the process of reduction is solely dependent on the presence of the metal named in the cathode-chamber. It is, moreover, immaterial whether a salt of copper or metallic copper in a finely-divided condition is added to the electrolyte in the cathode-chamber. In both cases the reaction proceeds over or by way of the regenerated or recovered metal or metal ion. There exists in the two cases merely a difference in the form of regeneration of the metal or metal salt, which form is governed by the magnitude of the electrolytic dissolving tension of the element in question. If the same is small, as in the case of copper, under the present invention, the metal after having entered the ion condition, and thus carried out the reduction, is directly restored to the metallic from the ion condition.

I will now describe my invention in detail with the aid of two examples embodying what I consider the preferable method of carrying the same into effect.

1. Reduction of nitrobenzene by the addition of cuprous chlorid to the cathode electrolyte.—An electrolytic element or battery is divided into a cathode and an anode chamber by a suitable diaphragm. The electrolyte of the anode-space consists of a ten-per-cent. solution of sulfuric acid, the anode consisting of any suitable indifferent metal. The cathode-chamber contains a cylindrical platinum electrode and is charged with a mixture of five hundred parts, by weight, of fuming hydrochloric acid, five hundred parts, by weight, of water, one hundred and twenty-three parts, by weight, nitrobenzene, and thirty parts, by weight, of cuprous chlorid. The nitrobenzene is maintained is suspension by a rapidly-revolving agitator or stirrer and when consumed is replaced by a fresh supply, thus making the process continuous. During the course of the process the mixture is preferably cooled. The current introduced has a density of about nineteen hundred amperes per square meter, and this current is maintained until regular liberation of hydrogen takes place and becomes visible. After the process of electrolysis is completed the copper of the cuprous chlorid will be found to have been separated in the form of fine metallic sponge, and no copper will be found in the cathode electrolyte. The nitrobenzene will be converted almost quantitatively into anilin chlorid, from which the base may be isolated by well-known methods.

2. Reduction of nitrobenzene by addition of copper-powder to the cathode electrolyte.—The process is carried out in the same manner as under Example 1, with the difference that instead of thirty grams of cuprous chlorid twenty grams finely-divided metallic copper are added to the cathode electrolyte.

What I claim, and desire to secure by Letters Patent of the United States, is—

1. The process of reduction which consists

in introducing a reducible substance into the acid electrolyte contained in the cathode-spaces of an electrolytic cell and passing a current through the same in the presence of copper, the electrolyte being one capable of reducing the copper to the ion condition.

2. The process of reducing a nitro compound which consists in introducing such compound into the acid electrolyte contained in the cathode-spaces of an electrolytic cell and passing a current through the same in the presence of copper, the electrolyte being one capable of reducing the copper to the ion condition.

3. The process of reduction which consists in introducing a reducible substance into the acid electrolyte contained in the cathode-space of an electrolytic cell and passing a current through the same in the presence of a copper cathode, the electrolyte being one capable of reducing the copper to the ion condition.

4. The process of reducing a nitro compound which consists in introducing such compound into the acid electrolyte of the cathode-space of an electrolytic cell and passing an electric current through the same in the presence of a copper cathode, the electrolyte being one capable of reducing the copper to the ion condition.

5. The process of reduction which consists in introducing a reducible substance into an acid cathode-bath containing a compound of copper and passing an electric current through said bath, the electrolyte being one capable of reducing the copper to the ion condition.

6. The process of reduction which consists in introducing a reducible substance into an acid cathode electrolyte and passing an electric current through the same in the presence of copper and at the same time stirring and cooling, the electrolyte being one capable of reducing the copper to the ion condition.

7. The process which consists in introducing a substance which is reducible to an amin into an acid cathode-bath in which is arranged a copper cathode and passing an electric current through the same.

8. The process which consists in introducing a substance which is reducible to an amin into an alcoholic-acid cathode-bath in which is arranged a copper cathode and passing an electric current through the same while stirring and cooling.

9. The process which consists in introducing a nitro compound to be reduced into a cathode-bath containing hydrochloric acid and in which is arranged a copper cathode to the action of an electric current.

10. The process which consists in introducing a nitro compound into a cathode-bath containing a cuprous compound and hydrochloric acid and having a cathode arranged therein and passing an electric current therethrough.

11. The process which consists in introducing a nitro compound into a cathode-bath containing hydrochloric acid and a cuprous salt and having a cathode arranged therein, and passing an electric current therethrough while stirring and cooling.

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

MAX BUCHNER.

Witnesses:
JACOB ADRIAN,
FRITZ ACH.