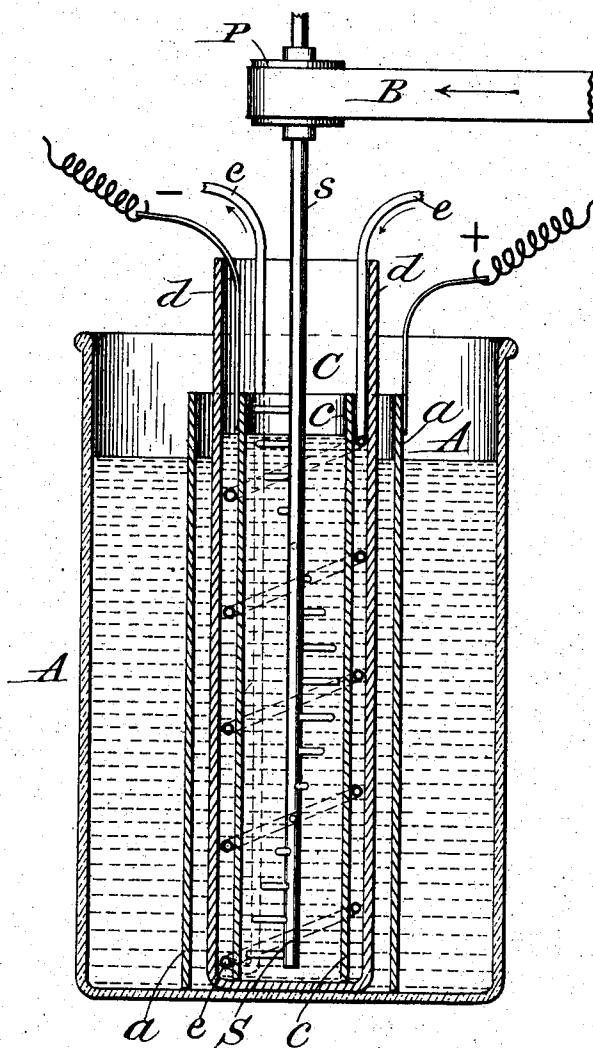


No. 761,284.

PATENTED MAY 31, 1904.

M. BUCHNER.  
REDUCTION OF NITRO-COMPOUNDS.  
APPLICATION FILED DEC. 13, 1902.

NO MODEL.



Witnesses

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384

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# UNITED STATES PATENT OFFICE.

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## REDUCTION OF NITRO COMPOUNDS.

SPECIFICATION forming part of Letters Patent No. 761,284, dated May 31, 1904.

Original application filed September 24, 1900, Serial No. 30,969. Divided and this application filed December 13, 1902. Serial No. 135,107. (No specimens.)

*To all whom it may concern:*

Be it known that I, MAX BUCHNER, a citizen of Germany, residing at Mannheim, Germany, have invented certain new and useful Improvements in the Reduction of Nitro Compounds; (originally described in my application, Serial No. 30,969, filed September 24, 1900, on which United States Patent No. 736,205 was granted, of which this is a divisional application;) 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 Letters Patent of the United States No. 700,670, dated May 20, 1902, I have described a process of electrolytic reduction of nitro compounds which consists, essentially, in causing an electric current to act on 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 lead; and my invention consists in the reduction of nitro compounds to amines by causing the electric current to pass through such compounds in acid solution and in the presence of the above metal or its 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 Patent No. 700,670 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 the finely-divided metal or a salt of such metal 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.

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

1. *Reduction of nitrobenzene to anilin by the addition of lead chlorid to the cathode-electrolyte.*—An electrolytic cell 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 conductor. 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, of nitrobenzene, and fifty parts, by weight, of lead chlorid. The nitrobenzene is maintained in 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 millimeter, and this current is maintained until regular liberation of hydrogen takes place and becomes visible. After the process of electrolysis is completed the lead of the lead chlorid will be found to have been separated in the form of fine metallic sponge and no lead will be found in the cathode-electrolyte.

2. *Reduction of nitrobenzene to anilin by addition of metallic lead to the cathode-electrolyte.*—The process is carried out substantially the same as under 1, with the difference that instead of fifty parts of lead chlorid forty parts of finely-divided metallic lead are added to the cathode-electrolyte.

In the accompanying drawing I have represented one out of many of the possible forms of apparatus and electrolytic cells for carrying out the process involved in my invention. In this drawing, A represents the anode-cham-

ber containing the anode-electrolyte, and *a* the anode; C, the cathode-chamber containing the cathode-electrolyte, and *c* the cathode, while *d* represents a diaphragm of any suitable material for separation of two electrolytes, as will be readily understood. S represents a suitable stirrer arranged within the cathode-electrolyte, the spindle or shaft *s* of which may be connected with any suitable source of power by a belt or pulley B P, as shown, or otherwise.

The cooling of the cathode-compartment may be carried out in any well-known manner, such as by cooling-coils *e* or the like.

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 substance to be reduced into the hydrochloric-acid electrolyte contained in the cathode-compartment of an electrolytic cell and passing a current through the same in the presence of lead.

2. The process of reducing a nitro compound which consists in introducing such compound into the acid electrolyte in the cathode-compartment of an electrolytic cell and passing a current through the same in the presence of lead.

3. The process of reduction which consists in introducing the substance to be reduced into the hydrochloric-acid electrolyte contained in the cathode-space of an electrolytic cell and passing a current through the same in the presence of a lead cathode.

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 lead cathode.

5. The process of reduction which consists

in introducing the substance to be reduced into a cathode-bath containing a compound of lead and hydrochloric acid and passing an electric current through said bath.

6. The process of reduction which consists in introducing the substance to be reduced into a hydrochloric-acid cathode-electrolyte and passing an electrolytic current through the same in the presence of lead, and at the same time stirring and cooling.

7. The process of producing amines which consists in introducing the corresponding nitro body into an acid cathode-bath in which is arranged a lead cathode and passing an electric current through the same.

8. The process which consists in passing an electric current through a nitro compound while in an acid electrolyte in the cathode-compartment of an electrolytic cell and in the presence of lead.

9. The process which consists in passing an electric current through a nitro compound while in an acid electrolyte in the cathode-compartment of an electrolytic cell and in the presence of a lead cathode.

10. The process which consists in passing an electric current through a substance which is reducible to an amine while in acid cathode-bath in which is arranged a lead cathode.

11. The process which consists in passing an electric current through a nitro compound while in an acid cathode-electrolyte and in the presence of lead, and at the same time stirring and cooling.

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

MAX BUCHNER.

Witnesses:

JACOB ADRIAN,  
H. W. HARRIS.