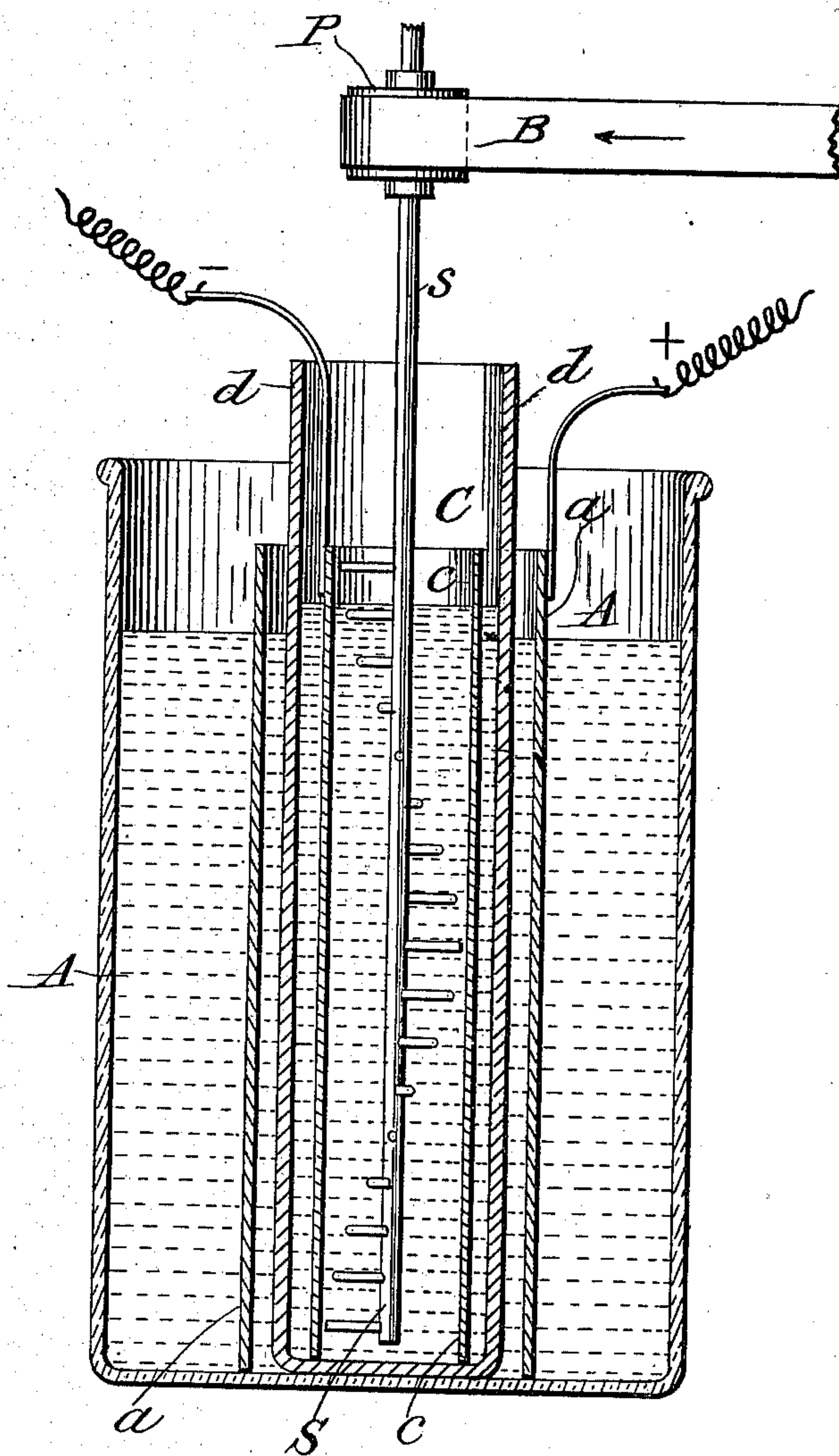


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M. BUCHNER.
REDUCTION OF NITRO COMPOUNDS.
APPLICATION FILED SEPT. 24, 1900.

NO MODEL.



WITNESSES:

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REDUCTION OF NITRO COMPOUNDS.

SPECIFICATION forming part of Letters Patent No. 736,205, dated August 11, 1903.

Application filed September 24, 1900. Serial No. 30,969. (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 Letters Patent of the United States No. 700,670, May 20, 1902, 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 lead, mercury, chromium, or iron, and my 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 ions of the above metals 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 metal in a finely-divided condition 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 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-solution tension of the element in question. If the same is small, the metal after having entered the ion condition and thus carried out the reduction is directly restored to the metallic from the ion condition. If, on the other hand, the same is great, the metal is not regenerated, but only the ion of a lower valency—for example, in the case of iron the Fe''' of the ferric chlorid can only be regenerated to the ferrous condition of Fe'' in the lower chlorid.

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 by the addition of lead 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-five parts, by weight, 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 meter, 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 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.

3. *Reduction of nitrobenzene by addition of ferrous chlorid to the cathode electrolyte.*—

- 5 The conditions and proportions of the ingredients of the bath-reducing salt and the substance to be reduced are the same as under Example 1 in this application and in my Patent No. 700,672, May 20, 1902. The current
10 density is also the same. In this case, however, no metallic iron, but only ferrous chlorid, is regenerated.

When employing the other metals hereinabove referred to, the conditions and proportions and the amperage per unit of surface
15 remain substantially the same, the proportions being somewhat modified, of course, where the metal is used instead of its salt, as indicated in Examples 1 and 2.

- 20 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.

25 In the drawing, A represents the anode-chamber, 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
30 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
35 suitable source of power by a belt and 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 and the like.

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

1. The process of reduction which consists in introducing the reducible substance into a cathode electrolyte of hydrochloric acid and
45 passing an electric current through the same in the presence of chromium.

2. The process of reduction which consists in introducing the substance to be reduced into a cathode-bath containing hydrochloric
50 acid and a compound of chromium, and passing an electric current through the same.

3. The process of reduction which consists in introducing the reducible substance into a cathode electrolyte of hydrochloric acid, and
55 passing an electric current through the same in the presence of chromium, and, at the same time stirring and cooling.

4. The process of reduction which consists in introducing a substance reducible to an amin into a cathode-bath containing hydro- 60 chloric acid and passing a current through the same in the presence of chromium.

5. The process of reduction which consists in introducing a substance reducible to an amin into a cathode-bath containing hydro- 65 chloric acid and passing a current through the same in the presence of a chromium cathode and at the same time stirring and cooling.

6. The process of reduction which consists in introducing a substance to be reduced into 70 the electrolyte in the cathode-compartment of an electrolytic cell and passing a current through the same in the presence of chromium.

7. The process of reducing a nitro compound which consists in introducing such compound into the electrolyte in the cathode- 75 compartment of an electrolytic cell and passing a current through the same in the presence of chromium. 80

8. The process of reduction which consists in introducing the substance to be reduced into the electrolyte contained in the cathode- 85 space of an electrolytic cell and passing a current through the same in the presence of a chromium cathode.

9. The process of reducing a nitro compound which consists in introducing such compound into the electrolyte of the cathode- 90 space of an electrolytic cell and passing an electric current through the same in the presence of a chromium cathode.

10. The process of reduction which consists in introducing the substance to be reduced into a cathode-bath containing a compound 95 of chromium and passing an electric current through said bath.

11. The process of reduction which consists in introducing the substance to be reduced into the cathode electrolyte and passing an 100 electric current through the same in the presence of chromium and at the same time stirring and cooling.

12. The process which consists in introducing a substance which is reducible to an amin into an acid cathode-bath in which is ar- 105 ranged a chromium cathode and passing an electric current through the same.

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

MAX BUCHNER.

Witnesses:

JACOB ADRIAN,
FRITZ ACH.