

GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

Specification No. 99181. Application No. 99181, dated 26th April 1965. Complete—
Specification left on 27th November 1965. (Application accepted 9th December 1966.)

Index at acceptance—32F2C [IX(1)].

IMPROVEMENTS IN OR RELATING TO THE ELECTROLYTIC REDUCTION OF NITROGUANIDINE
TO AMINOGUANIDINE.

PROVISIONAL SPECIFICATION.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN
REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNA UDUPA, GOBICHETTIPALAYAM SRINIVASAN
SUBRAMANIAN AND KODETHOOR SHRIVARA UDUPA, ALL OF CENTRAL ELECTROCHEMICAL RESEARCH
INSTITUTE, KARAUKUDI-3, S. RLY., MADRAS STATE, INDIA, ALL INDIAN CITIZENS.

The following specification describes the nature of this invention.

This invention relates to improvements in or relating to the electrolytic reduction of nitroguanidine to aminoguanidine.

Hitherto it has been the practice (a) to reduce nitroguanidine in a neutral medium with ammonium chloride and zinc dust or (b) electrolytically reduce in various ways using stationary cathodes.

This is open to objection in that in (a) the reduction efficiency is somewhat low and the isolation of the final reduction product is a rather tedious process and in (b) low current efficiency is obtained and low current densities only can be employed.

The object of this invention is to obviate these disadvantages by reducing the nitroguanidine electrochemically using a rotating amalgamated cathode.

To these ends, the invention broadly consists in reducing the nitroguanidine in sulphuric acid or ammonium sulphate solution but preferably the latter in a 30% concentration using a rotating amalgamated lead cathode. The reduction is carried out at low temperature ranges between 5°C to 20°C but preferably at 10°C using current densities upto 20 amp/dm² but preferably 10 amp/dm². After the electrolysis is over, the catholyte is treated with sodium bicarbonate (70 g per litre) and kept overnight in a refrigerator. The aminoguanidine bicarbonate which separates as a fine crystalline powder is filtered, washed and dried.

The following typical examples are given to illustrate the invention :

Example 1

Catholyte	150 cc of 30% ammonium sulphate solution.
Anolyte	20 cc of 10% sulphuric acid.
Amount of nitroguanidine taken	2 g.
Current density	15 amp/dm ² .
Temperature	10-12°C
Cathode	Rotating amalgamated lead (Effective area = 0.3 sq. dm).
Anode	Lead.

Aminoguanidine estimated (as free base)	1.23 g.
Current efficiency	63.8%
Yield	86.6%

Example 2

Catholyte	150 cc of 30% ammonium sulphate solution.
Anolyte	20 cc of 10% sulphuric acid.
Amount of nitroguanidine taken	6 g.
Current density	10 amp/dm ² .
Temperature	10-12°C.
Cathode	Rotating amalgamated lead (Area=0.3 sq. dm.)
Anode	Lead.
Aminoguanidine estimated (as free base)	3.86 g.
Current efficiency	73.6%
Yield	90.6%

The following are among the main advantages of the invention :

1. The use of a rotating cathode simplifies the design of high amperage cell and permits use of high current densities.
2. Under these conditions, the electrolytic reduction of nitroguanidine to aminoguanidine proceeds smoothly and high yields are obtained without an adverse effect on the current efficiency.
3. Both the material yield and the current efficiency are high using rotating cathode as against the reported high yield and low current efficiency of reduction using a stationary cathode.

R. BHASKER PAI

Patent Officer,

COUNCIL OF SCIENTIFIC AND INDUSTRIAL
RESEARCH.

Dated this 17th day of April 1965.

COMPLETE SPECIFICATION.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN
INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNA UDUPA, GOBICHETTIPALAYAM SRINIVASAN
SUBRAMANIAN AND KODETHOOR SHRIVARA UDUPA, ALL OF THE CENTRAL ELECTRO-
CHEMICAL RESEARCH INSTITUTE, KARAUKUDI-3, S. RLY., INDIA, ALL INDIAN CITIZENS.

The following specification particularly describes and ascertains the nature of this invention and the manner
in which it is to be performed.

This invention relates to improvements in or relating to the electrolytic reduction of nitroguanidine to aminoguanidine.

Hitherto it has been the practice (a) to reduce nitroguanidine in neutral medium with ammonium chloride

and zinc dust or (b) electrolytically reduce it in various ways using stationary cathodes.

This is open to objection in that in (a) the reduction efficiency is somewhat low and the isolation of the final reduction product is a rather tedious process and in

Price : TWO RUPEES.

(b) low current efficiency is obtained and low current densities only can be employed.

The object of this invention is to obviate these disadvantages by reducing the nitroguanidine electrochemically by using a rotating cathode, preferably of lead, zinc, amalgamated lead.

The invented process for the electrolytic reduction of nitroguanidine to aminoguanidine consists in reducing the nitroguanidine in sulphuric acid or ammonium sulphate solution but preferably the latter up to a 30% concentration preferably between 10-12°C and using current densities up to 20 amp/dm² but preferably at 10 amp/dm² using a rotating cathode of lead, zinc or amalgamated lead.

The reduction is carried out in a divided cell using a diaphragm, preferably of ceramic porous pot in which anolyte is taken, preferably 20% sulphuric acid and a perforated anode, preferably of lead or antimonial lead or lead-silver alloy.

The aminoguanidine that is obtained in the cell is recovered as bicarbonate salt by precipitating the catholyte with carbon dioxide or ammonium carbonate or sodium bicarbonate.

Thus, the reduction may be carried out at low temperature ranges between 5°C and 20°C but preferably at 10-12°C using current densities upto 20 amp/dm² but preferably 10 amp/dm². After the electrolysis is over, the catholyte is neutralised with carbon dioxide or ammonium carbonate or sodium bicarbonate till the pH is 8 and on keeping at a temperature of about 5 to 10°C, aminoguanidine bicarbonate separates out as a fine crystalline powder and is filtered, washed and dried. The mother liquor can be reused again when the precipitation is carried out with carbon dioxide or ammonium carbonate.

The following typical examples are given to illustrate the invention :

Example I

Catholyte	2500 ml of 30% ammonium sulphate solution.
Anolyte	800 ml of 20% sulphuric acid.
Amount of nitroguanidine taken	50 g.
Current density	10 amp/dm ² (current 20 amp).
Temperature	10-12°C.
Cathode	Rotating lead-effective area = 1.5 dm ² .
Anode	Perforated lead.
Aminoguanidine recovered as bicarbonate	36 g.
Current efficiency	62.5%.
Yield	75%.
Cell voltage	4.5-5 volts.
Energy consumption	10.5 kwh/kg.

Example II

Catholyte	2500 ml of 30% ammonium sulphate solution.
Anolyte	800 ml of 20% sulphuric acid.
Amount of nitroguanidine taken	120 g.
Current density	10 amp/dm ² .
Temperature	10-12°C.
Cathode	Rotating zinc cathode (Area = 1.5 dm ²).
Anode	Lead.
Aminoguanidine bicarbonate recovered	90 g.
Current efficiency	80%.
Yield	90.3%.
Cell voltage	4.5 to 5 volts.
Energy consumption	9.4 kwh/kg.
Loss in weight of zinc cathode	Less than 1% (0.90).

We claim :

1. A process for the electrolytic reduction of nitroguanidine to aminoguanidine which consists in reducing the nitroguanidine in sulphuric acid or ammonium sulphate solution but preferably the latter up to a 30% concentration preferably between 10-12°C and using current densities upto 15 amp/dm² but preferably at 10 amp/dm² using a rotating cathode of lead, zinc or amalgamated lead.

2. A process for the electrolytic reduction of nitroguanidine as claimed in Claim 1 wherein the reduction is carried out in a divided cell using a diaphragm, preferably of ceramic porous pot in which anolyte is taken, preferably 20% sulphuric acid as anolyte, and a perforated anode, preferably of lead or antimonial lead or lead-silver alloy.

3. A process as claimed in Claim 1 or 2 wherein the aminoguanidine that is obtained in the cell is recovered as bicarbonate salt by precipitating the catholyte with carbon dioxide or ammonium carbonate or sodium bicarbonate.

4. A process for the electrolytic reduction of nitroguanidine to aminoguanidine substantially as hereinbefore described.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

Dated this 15th day of November 1963.