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IMPROVEMENTS IN OR RELATING TO THE ELECTROCHEMICAL PRODUCTION OF O-AMINO  
TOLUENE FROM ORTHONITRO TOLUENE

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).

The following specification describes the nature of this invention.

This is an invention by Handady Venkatakrishna Udupa. Scientist: Mysore Seshaiyer Venkatachalapthy, Scientist and Sankaranarayanaier Chidambaram, Senior Scientific Assistant, all of Central Electrochemical Research Institute, Karaikudi-3, Madras State, India, all Indians.

This invention relates to improvements in or relating to the electrochemical preparation of Ortho amino toluene.

Hitherto it has been proposed to reduce O-nitro toluene by chemical reducing agents like iron powder and zinc and electrolytically by employing stationary cathodes for the preparation O-amino toluene from O-nitro toluene.

This is open to the objection that the processes reported in literature suffer from the following drawbacks.

(i) It is rather tedious and laborious to separate the product from the reactants while employing chemical reducing agents.

(ii) The choice of proper cathode to increase the amine content of the product.

(iii) Only low current densities can be employed at a stationary cathode.

(iv) Information available on the cathodic preparation of O-amino toluene is not adequate enough for setting up a practical scale unit due to the lack of the design of high amperage cells.

The object of this invention is to obviate these disadvantages by the improvements now effected in the process by us.

According to the present invention, chemical reducing agents are avoided by using either a rotating or a stationary cathode for the reduction of O-nitro toluene to produce O-amino toluene. The present process is superior to the chemical processes hitherto described in literature since it not only eliminates the tedious and laborious process of separating the product from the reactants but also gives a very pure product and thereby renders the process more easy to operate without economic drawbacks. Additional advantage in the process now established by us is that by using a rotating cathode, high current density could be employed without adversely affecting the energy economics of the process.

To these ends, the invention broadly consists in reducing cathodically O-nitrotoluene to give O-amino toluene in an electrolytic cell, fitted with either a rotating tin, or tinned or tin plated cathode. The catholyte was separated from anolyte by means of a porous diaphragm. A 35% solution of sulphuric acid was used as anolyte and the anode was made of lead or lead alloy. The catholyte employed was hydrochloric acid of density 1.03 and the concentration of the depolariser was kept between 10 & 30% but a solution of 20% could give the maximum current efficiency and yield. A current density upto 10 amp/dm<sup>2</sup> in the case of stationary

cathode and 10—25 amp/dm<sup>2</sup> with a rotating cathode could be employed. The temperature of the catholyte could vary from 30—50°C but a temperature of 40—45°C was employed for most of the experiments. A current efficiency of 85% and an yield of 86% was obtained.

The following typical examples are given to illustrate the invention.

Example I

Electrolytic reduction of O-nitro toluene using a stationary tin cathode.

128 g of o-nitro toluene was taken in 1200 cc of hydrochloric acid of density 1.03. Tin was employed as cathode. 35% aqueous solution of sulphuric acid was employed as anolyte and lead was used as anode. The catholyte was separated from the anolyte by means of a ceramic diaphragm. During the electrolysis, the temperature of the catholyte was kept between 40—45°C by means of a water bath. A current density of 10 amp/dm<sup>2</sup> was employed. After passing 145 amp hrs 80 g of O-amino toluene was obtained with a current efficiency of 84% and an isolated yield of 75—80%. The energy consumption comes to 10.8 kwh/kg.

Example II

Electrolytic reduction of O-nitro toluene using a rotating tin cathode:

414 g of O-nitro toluene was taken 2000 cc of hydrochloric acid of density 1.03. A rotating disc of tinned copper was employed as cathode. The r.p.m. was kept between 1500—2000. The average cell voltage was 5.0V. During electrolysis, the temperature of the catholyte was kept between 40° & 45°C. A current density of 20 amp/dm<sup>2</sup> was employed and after passing 495 amp-hr 280 g of O-amino toluene was obtained. No unreduced nitro-compound could be detected after electrolysis. A current efficiency of 85% and an isolated yield of 87% was obtained. The anode employed was the same as that given in example I. The energy consumption comes to 9.0 kwh/kg.

The following are among the main advantages of the invention:

(1) The process does not involve the use of any chemical reducing agents and as such its removal does not arise.

(2) Isolation of the product is simplified, thereby also giving a product which is pure.

(3) The employment of a rotating cathode would very much help in the design of high amperage cells in order to adopt the process for large scale production.

Dated this 13th day of December, 1967.

Sd.

PATENTS OFFICER,

Council of Scientific & Industrial Research.

## Complete Specification

### IMPROVEMENTS IN OR RELATING TO THE ELECTROCHEMICAL PRODUCTION OF O-AMINO TOLUENE FROM ORTHO NITRO TOLUENE

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New-Delhi-1, India, an Indian register body incorporated under the Registration of Societies Act (Act XXI of 1860).

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

This is an invention by HĀNDADY VENKATAKRISHNA UDUPA, Scientist, MYSORE SESHAIYER VENKATACHALAPATHY, Scientist and SANKARANARAYANAIYER CHIDAMBĀRAM, Scientist, all of the Central Electrochemical Research Institute, Karaikudi-3, Madras State, India, all Indian citizens.

This invention relates to a improvements in or relating to the electrochemical production of O-amino toluene from Ortho nitro toluene.

The annotated title of the invention, to clearly indicate the field of industry to which the invention relates:

O-Amino toluene is prepared by electrochemical reduction of O-nitro toluene. The invention relates to the field of organic chemical industry with special reference to intermediates for dyestuffs industries.

Prior knowledge:

Hitherto it has been proposed to reduce ortho nitro toluene by chemical reducing agents like iron powder and zinc and electrolytically employing stationary cathodes.

Drawbacks connected with hitherto known processes:

The process hitherto reported in literature suffers from the following drawbacks: (i) the choice of a proper cathode to increase the amine content of the product (ii) it is rather tedious and laborious to separate the product from the reactants while employing chemical reducing agents.

The main object of the invention:

The main object of the invention is to obviate the drawbacks by adopting electrochemical reduction technique using rotating cathode, and by a proper choice of cathode material.

The main finding underlying the invention:

According to the present invention chemical reducing agents are avoided by using either a rotating or a stationary cathode for the electrolytic reduction of ortho nitro toluene to ortho-amino toluene.

The present process is superior to the processes hitherto described in literature since it not only eliminates the tedious process of separating the product from the reactants but also gives a very pure product. Thereby it renders the process more easy to operate. Additional advantage in the process now established by us is that by using a rotating cathode high current density would be employed. The process is more economical when compared to conventional method since only electrical energy is consumed by the product of O-amino toluene (9 kwh/kg) of O-amino toluene).

According to the present invention, the process consists in the electrochemical preparation of O-amino toluene from ortho nitro toluene using tin or tin plated cathode either stationary rotating and having an anode of lead which has been separated from the cathode by means of a porous diaphragm.

Lead clad copper may be used as anode. Hydrochloric acid may be used as catholyte and the percentage may be varied between 4 and 6%.

The concentration of sulphuric acid in the anolyte could be varied between 5 and 10% but preferably 5%.

As stated, the cathode may be stationary or rotating, and while stationary an auxiliary stirrer is employed.

A porous diaphragm material, preferably of porous ceramic is used to separate the cathode chamber from the anode chamber.

Thus, ortho nitro toluene was electrolytically reduced to give O-amino toluene in a cell fitted with a rotating tin or tinplated cathode. The catholyte was separated from anolyte by means of a porous diaphragm. The concentration of the depolariser was kept between 10 and 30 percent in dilute hydrochloric acid electrolyte. A current density upto 10 amp/dm<sup>2</sup> in the case of stationary cathode and 10—30 amp/dm<sup>2</sup> with a rotating cathode could be employed. The temperature of electrolysis could vary from 45—50°C but a temperature of 43—45°C was employed for most of the experiments. A current efficiency of 85% and an yield of 86% was obtained.

Typical examples:

#### Example I

Electrolytic reduction of Ortho nitro toluene using stationary cathode.

128 gm of ortho nitro toluene was taken 1200 ml of 6% hydrochloric acid. Tin was employed as cathode. Lead clad copper rod was used as anodes. During the electrolysis, the temperature of the electrolyte was kept between 35—40°C by means of a waterbath. A current density of 10 amp/dm<sup>2</sup> was employed. After passing 145 amp-hrs 80 g. of ortho amino toluene was obtained with a current efficiency of 84% and an isolated yield of 75—80%. The energy consumption comes to 10.8 kwh/kg. An auxiliary stirrer was included in the cell. The average cell voltage was 5.5V. The anolyte employed was 5% sulphuric acid. A porous pot was used to separate the anolyte from the catholyte.

#### Example II

Electrolytic reduction of Ortho nitro toluene using a rotating cathode

414 g of ortho nitro toluene was taken in 2000 ml of 5% hydrochloric acid. A rotating disc of tinned copper was employed as cathode. The r.p.m. was kept between 1000 and 1200. During the electrolysis, the temperature of the electrolyte was kept between 40 and 45. A current density of 20 amp/dm<sup>2</sup> was employed and after passing 495 amp-hrs, 280 g. of O-amino toluene was isolated. No un-reduced O-nitro toluene could be detected. A current efficiency of 85% and an isolated yield of 87% was obtained. The anode employed was the same as given in Example I. The energy consumption comes to 9.0 kwh/kg and the average cell voltage was 5V. The anolyte employed was 5% sulphuric acid. A porous pot was used to separate the anolyte from the catholyte.

Summary:

A process has been invented for the electrolytic production of O-amino toluene from Ortho nitro toluene. The process eliminates the use of conventional methods which require further purification of the product. A high current density could be employed in the cell by the use of rotating cathode which renders the process more easy to operate.

We claim:

(1) A process for the electrochemical preparation of O-amino toluene from Ortho nitro toluene using tin or tin plated cathode either stationary or rotating and having an

anode or lead which has been separated from the cathode by means of a porous diaphragm.

(2) A process as claimed in claim (1) wherein lead clad copper are used as anodes.

(3) A process as claimed in claim (1) wherein hydrochloric acid is used as catholyte and the percentage could be varied between 4 and 6%.

(4) A process as claimed in claim (1) wherein a current density upto 10 amp/dm<sup>2</sup> in the case of stationary cathode and 10—30 amp/dm<sup>2</sup> in the case of rotating cathode are employed, but preferably 20 amp/dm<sup>2</sup>.

(5) A process as claimed in claim (1) wherein the concentration of sulphuric acid in the anolyte could be varied between 5 and 10% but preferably 5%.

(6) A process as claimed in claim (1) wherein the temperature range could be 40—50°C but preferably at 43—45°C.

(7) A process as claimed in claim (1) wherein the cathode is stationary or rotating and while stationary an auxiliary stirrer is employed.

(8) Ortho amino toluene whenever prepared from Oriho nitro toluene according to the process substantially as hereinbefore described.

(9) A process as claimed in claim (1) wherein the cathode chamber is separated from the anode chamber by the use of a porous diaphragm material, preferably of porous ceramic.

Dated this 9th day of October, 1968.

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