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" PROCESS FOR ELECTROCHEMICAL PREPARATION OF BETA
PHENYLETHYLAMINE USING COBALT BLACK CATHODE "

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registered body incorporated under the Registration
of Societies Act (Act XXI of 1960)

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

PRICE : TWO RUPEES

This is an invention by Handady Venkatakri^{shna} Udupa, Director; Venkatasubramanian Krishnan, Scientist and Arunachalam Muthukumaran, Junior Scientific Assistant - All of Central Electrochemical Research Institute, Karaikudi - 623 006, Tamil Nadu, India - all Indian citizens.

This invention relates to the electrochemical preparation of beta-phenylethylamine from benzyl cyanide by electroreduction technique using deposited cobalt black cathode over graphite substrate under stationary conditions. This amine can also be prepared by catalytic reduction of the cyanide using precious metal oxides. Electrochemical methods have already been developed for the preparation of this amine using deposited palladium black as described in Indian Patent No. 143906 and nickel black cathode as described in Indian Patent No.145304. The electrochemical method described here is expected to be less costlier than the catalytic method. Beta-phenyl-ethylamine is a drug intermediate used in the manufacture of variety of anti diabetic drug. It is also an efficient corrosion inhibitor.

The main object of the invention is to prepare beta-phenylethylamine using cobalt black cathode by an electrochemical method. This is the first time that such a deposited cobalt black cathode has been employed successfully on a pilot plant scale for the production of beta-phenylethylamine.

In an aqueous ethanolic ammonium sulphate medium, benzyl cyanide was reduced using deposited cobalt black cathode with satisfactory yield of beta-phenylethylamine.

The novelty of the process is mainly in the use of the deposited electrode for the first time on a pilot plant scale for the manufacture of beta phenylethylamine. The cost of production of the amine is now about twenty times reduced (approximately) compared with the cost of the amine using palladium black electrode.

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This invention consists of three stages. In the first cobalt black is deposited over graphite plate. In the second stage the electro-reduction of benzyl cyanide is carried out with the deposited electrodes in aqueous ethanolic ammonium sulphate medium. In the third stage, the amine is isolated in pure form from the catholyte.

In the first stage, cobalt black is deposited over graphite plate by electrodeposition from a bath containing cobalt ammonium sulphate.

In the second stage, the reduction of benzyl cyanide in aqueous ethanolic ammonium sulphate medium was carried out with cobalt black cathode. A ceramic porous pot, containing aqueous sulphuric acid as anolyte was used as the diaphragm into which a hollow lead silver alloy cylinder was placed as the anode. The temperature of the catholyte was maintained around 25°C. A current density of 5 amp/sq.dm was found to be the most advantageous, taking into account various factors of electrolysis. As the theoretical charge was found to be insufficient extra current was passed to obtain a maximum yield.

The third stage is the isolation of pure beta-phenylethylamine as follows: At the end of the electrolysis, the catholyte was distilled to recover alcohol when the unreduced benzyl cyanide was found to float which was removed. The residue in aqueous solution was then neutralised with NaOH, to liberate the amine. The amine was then purified by distilling at a boiling point of 198°C.

The following is the typical example to illustrate the process of the invention :

Example

Catholyte : 60 litres of ethanol + 52 litres
water + 3.6 kg of $(\text{NH}_4)_2\text{SO}_4$ + 3.6 kg

Anolyte	: Aqueous sulphuric acid 10% (16 litres)
Cathode	: Deposited cobalt black cathodes over graphite plate (16 nos of 20 cm x 15 cm size; effective area 6 sq. dm inclusive of both sides)
Anode	: 16 nos of hollow perforated cylinder of lead silver alloy (area of each anode is 5.5 sq. dm). These anodes are kept inside ceramic diaphragm
Electrolytic cell	: Circular PVC vessel to accommodate these 16 cathodes plates and 16 diaphragms
Current density employed for reduction	: 5 A/sq. dm
Current passed	: 500 amperes
Cell voltage	: 10-12V
Pure beta phenylethylamine obtained	: 2.238 kg
Yield	: 64%
Current efficiency	: 40%

The main advantage of the process of the invention is that the use of cobalt black cathode has brought down the cost of phenylethylamine considerably.

We Claim :

1. An improved process for the preparation of β -phenylethylamine by electrolytic reduction of benzylcyanide wherein a cobalt black deposited cathode is used in aqueous ethanolic ammonium sulphate solution and a lead silver alloy as anode and separating the β -phenylethylamine formed from the catholyte.
2. A process as claimed in claim 1 wherein the electrolysis is carried out in a diaphragm cell at a temperature upto 25°C at a current density of 5 amp/sq.dm.
3. A process as claimed in claims 1 or 2 wherein β -phenylethylamine is separated by distillation to recover the alcohol and unreduced benzyl cyanide and the residue is neutralised with excess of alkali to liberate the amine.

4. A process as claimed in any of the preceding claims wherein the cobalt black cathode used is a graphite plate deposited with cobalt black.

Dated this 11th day of April, 1979.

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