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A new preparation method of N-Pentamethylbenzamide

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A new preparation method of N-Pentamethylbenzamide

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ABSTRACT

Electrochemical preparation method of N-Pentamethylbenzamide was investigated

INTRODUCTION

The only preparation method of N-Pentamethylbenzamide was found in 1976 by J. Barry, A. Mayeda and C. Ross(1) and according to his results this compound was prepared by amidoalkylation of pentamethylbenzene, and metilen bis-benzamide was used as an electrophile.

In this work N-Pentamethylbenzylbenzamide was prepared electrochemically from Hexamethylbenzene.

EXPERIMENTAL

a) Apparatus:

In this work Hi-Tek potentiostat and Chemical Electronics waveform generator were used with coulometer. Conventional H shape electrolysis cell with Pt electrodes was used for the preparative experiment. Ag/Ag^{+}electrode was used as a reference electrode.

b) Experimental procedure:

A solution of 0,3 mM hexamethylbenzene and 0,4 M tetrabutylammoniumtetrafluoroborata (2) in benzonitrile was electrolysed at a 1,25 V (vs Ag/Ag^{+}) constant anode potential. During the experiment the amount of electricity was recorded by coulometer and i-Q plot was shown in, fig 1.

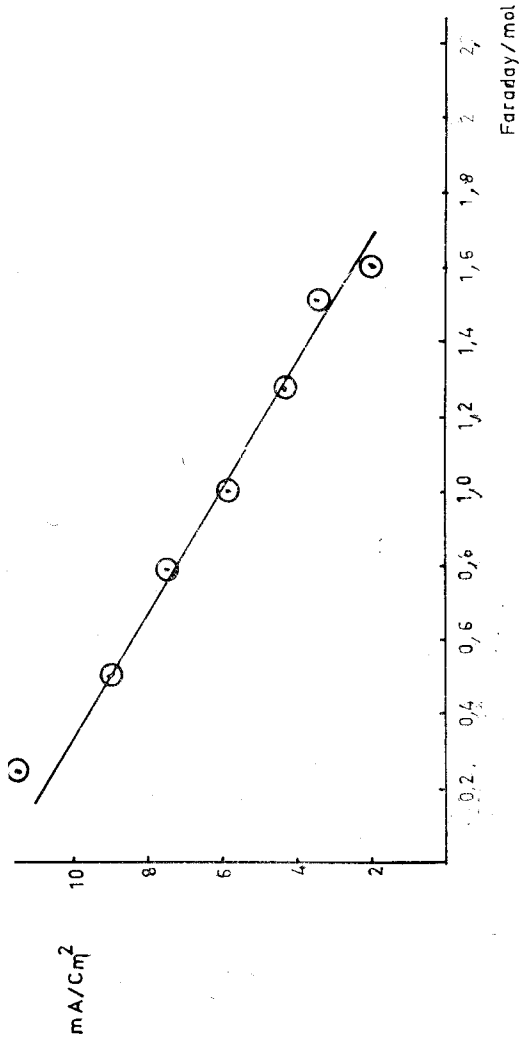

Fig. 1

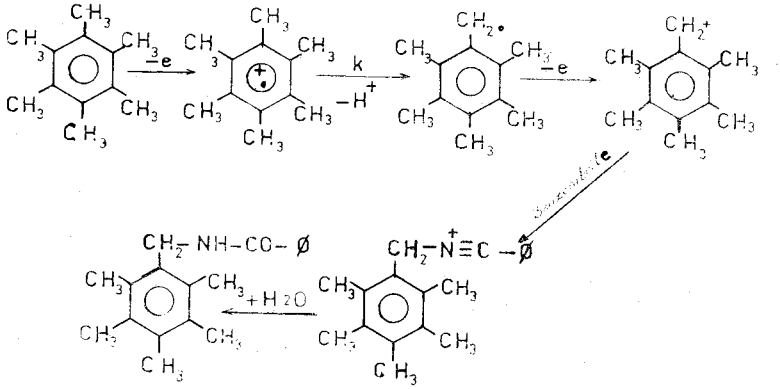

Fig. 2

After stopping the electrolysis, water was added into anolyte and aqueous phase was separated from benzonitrile. The organic solvent was removed in vacuo, and crude product extracted with ether and applied to alumina column.

Chloroform was used as an eluant after evaporation of eluent N-Pentamethylbenzylbenzamide crystallized from chloroform and analysed by GC-MS,IR and UV techniques. Mp: 195-198°C and yield 38%.

RESULT AND DISCUSSION

Electrochemical oxidation mechanism of Hexamethylbenzene in various solvents and conditions will be published later. According to this results Hexamethylbenzene gave 1 electron initially and become a radical cation, this species easly loose one of their proton and pentamethylbenzyl radical was formed. Further oxidation of this radical gave carbonium ion. Carbonium ion intermediate in benzonitrile gave a nitrilium ion and after quenching with water N-Pentamethylbenzylbenzamide was formed. The electrochemical oxidation mechanism was illustrated on Fig 2. From these results it can be easly seen that the overall reactons 2 electron oxidation of Hexamethylbenzene and this results is in good acoordance with the finding of i-Q plot.

Finally with this synthesis, it was shown that although the preparation of such monoamides by purely organic procedure is difficult, electrochemical procedure is promising.

There are of course, several factors which will greatly influence the yields of product. However this simple, single-step route to preparation of amides has considerable synthetic potential.

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2- **D.B. Clark, H.** *Fleischmann and D. Pletcher* J.C.S. Perkin II 1578 (1973).

ÖZET

Bu çalışmada N-Pentametilbenzamit'in elektrokimyasal yolla yapılan sentezi anlatılmıştır. Bu amaçla başlangıç maddesi olarak Hegzametilbenzen kullanılmış ve sentez kontrollu potansiyelde elektrokimyasal olarak yapılmıştır. Çalışma eletrodu olarak Pt elektrotlar ve çözücü olarak ta benzonitril kullanılmış ve hegzametilbenzenin elektrokimyasal yükseltgenme mekanizması açıklanmıştır.

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