ELECTROCHEMICAL REACTIONS IN THE PROCESS OF THE SYNTHESIS OF ANESTHESINE FROM TOLUENE

Magomedova Zalmo Magomedovna, Khidirov Shahabudin Shaidabekovich

Daghestan State University, Makhachkala, Daghestan, Russian Federation

This work is dedicated to the synthesis of the ethyl ether of $p$-aminobenzoic acid (anesthesine) from toluene. The process is achieved according to the following diagram:

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\begin{align*}
\text{CH}_3 & \quad \text{HNO}_3 \\
\text{CH}_3 & \quad \text{H}_2\text{SO}_4 \\
\text{NO}_2 & \quad [\text{O}] \\
\text{COOH} & \quad + \text{C}_2\text{H}_5\text{OH} \\
\text{COOC}_2\text{H}_5 & \quad [\text{H}] \\
\text{CO}_2\text{C}_2\text{H}_5 & \quad \text{NH}_2
\end{align*}
\]

It is known, that the aromatic nitrocompounds are well reduced electrochemically. In a number of cases the electrochemical method has advantages over the chemical one. Therefore stages 2 and 4 in the given diagram by us are studied by the electrochemical method. Measurements were conducted in the three-electrode electrochemical cell, both with the divided cathode and anode compartments, and without the separation. Potential is measured relative to chlorinesilver electrode and is converted according to the hydrogen scale.

In the volt-ampere curves, taken on the electrode from glass-carbon, 2 maximums of current are observed against the background of the aqueous solution of acetonitrile. They correspond to two stages of the electrochemical oxidation of $p$-nitrotoluene to $p$-nitrobenzoic acid. The first maximum appears in the region of potentials 1,4-1,5 v, and the second - 2,2 v. Oxidation occurs through the formation of intermediate product benzaldehyde, the presence of which we have discovered by micro-electrolysis with the controlled potential.

The process of reduction of the ethyl ether $p$-nitrobenzoic acid to the ethyl ether of $p$-aminobenzoic acid is studied in the acid medium. In the volt-ampere curve, obtained in the solution of ethyl ether $p$-nitrobenzoic acid two maximums of current are revealed with the potentials -0,6 V and -1,2V: the first one corresponds to the formation of the ethyl ether $p$-hydroxilaminobenzoic acid, and the second one - to its reduction up to the anesthesine.

On the basis of the data obtained the optimum conditions (concentration of parent substance, the material of the electrode, current density, temperature) are selected for the electrosynthesis of the ethyl ether of $p$-aminobenzoic acid (yield 90%).