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Electrochemical Version of the Knorr Reaction

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Abstract

Electrochemical reduction of ethyl 2-hydroxyimino-3-oxobutanoate in acetic acid in the presence of sodium acetate requires four electrons per molecule and yields an intermediate amino derivative, whose condensation with ethyl acetoacetate affords diethyl 2,4-dimethylpyrrol-3-5-dicarboxylate. The substance-based yields of the pyrrol are 74% at a steel cathode, 72% at a nickel cathode, 68% at a platinum cathode, 67% at a lead cathode, and 63% at a copper cathode. Electrolysis conditions: current density 120 mA/cm2; amount of electricity 161 A h per mol of the initial compound; 95°C. With an amount of electricity of 107 A h per mol of the initial compound, the yield of diethyl 2,4-dimethylpyrrole-3,5-dicarboxylate at a steel cathode increases from 37 to 73% in the temperature range from 40 to 105°C. The process can be regarded as an electrochemical version of the Knorr synthesis.