Pulsed Fe Electro-Oxidation for Catalytic Synthesis of Hydantoin Derivatives

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This paper presents an original and practical organic electrochemical/chemical (EC/C) synthesis to prepare hydantoin derivatives from N-Alkyl-piperidin-4-ones. The electrochemical assisted process is an alternative to the traditional Bucherer-Bergs method. The main contribution is the *in-situ* production of Fe (III), by pulsed electro-oxidation of an iron wire, in a methanol-water (1:1) mixture at 58°C. The oxidized iron, on the wire surface, served as catalyst in the traditional synthetic path of hydantoin derivatives, and the KCN and (NH₄)₂CO₃ aided as supporting electrolyte for the electrolytic process. EC/C prepared hydantoins were characterized by IR, NMR (¹H, ¹³C), and MS. The pulsed potential applied to the iron wire was 0.9V/Ag/AgCl/Cl⁻_{sat} KCl(MeOH) during 300s, alternated with -0.1V/Ag/AgCl/Cl⁻_{sat} KCl(MeOH) during 60s. The electrode potential program attempted to avoid electrode surface passivation. After identifying the produced hydantoins by a thin-layer chromatography, electric charge corresponding to the current integration during the pulses of 300 s (10 cycles) gave a total oxidation charge density of 5.57C/cm² for hydantoin 8-(3-nitrobenzyl(-1,3,8-triazaespyrodecane-2,4-dione) and 2.68C/cm² for 8-(4-nitrobenzyl(-1,3,8-triazaespyrodecane-2,4-dione)). The EC/C synthesis was compared with the traditional Bucherer-Berg method, resulting in an increase of 12-fold more hydantoins in 60 min.

Keywords: hydantoins, N-piperidone, Fe electro-oxidation in methanol-water, organic electrochemical/chemical synthesis.

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