HOMOLYTIC ALKYLATION OF 2-METHYLQUINOLINE WITH 18-CROWN-6

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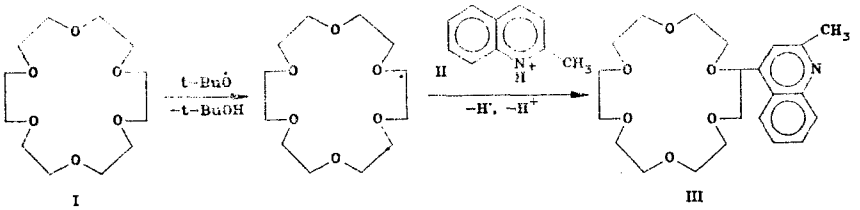
The described methods for the synthesis of crown ethers containing a quinoline substituent are distinguished by their multistage nature and low yield [1, 2]. We have effected a homolytic alkylation of 2-methyl-quinoline (II) by 18-crown-6 (I) and compound (III) was obtained in 78% yield on the crown ether (I) which had reacted.



I III

The conversion of crown ether (I) and 2-methylquinoline (II) was 75-90%. The oxidation—reduction system tert—butyl hydroperoxide—divalent iron sulfate served as a source of tert-butoxyl radicals. Reaction was carried out by the modified procedure of [3] by adding tert-butyl hydroperoxide (2×10^{-2} mole) to a solution of crown ether (I) (10^{-2} mole), (II) sulfate (10^{-2} mole), and iron sulfate (0.5×10^{-2} mole) in dimethylsulfoxide (DMSO) for 1 h in an atmosphere of argon at room temperature. Reaction product (III) was separated by liquid column chromatography (Al_2O_3, eluent was a mixture of hexane with chloroform, 5:1). The structure of the reaction product was shown by PMR and ^{13}C NMR spectra and by data of elemental analysis.

(2-Methylquinolin-4-yl)-18-crown-6 (III). PMR spectrum, δ, ppm (in $CDCl_3$): 2.57 s (3 H, CH_3), 3.40-3.70 m (22 H, CH_2—O), 5.27 t (1 H, CH—O), 7.16-8.08 m (5 H, Ar). ^{13}C NMR spectrum, δ, ppm (in $CHCl_3$): 25.0 q (1 C, CH_3), 68.7-78.0 m (11 C, CH_2-O), 78.5 d (1-C, CH—O), 119.5-158.3 m (9-C, Ar). Found: C 66.0; H 8.0; N 4.2%. $C_{22}H_{31}NO_6$. Calculated: C 65.2; H 7.7; N 3.5%.

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