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ELECTROCHEMICAL SYNTHESIS OF NEW 2-(2'-FURYL)BENZ-IMIDAZOLE DERIVATIVES

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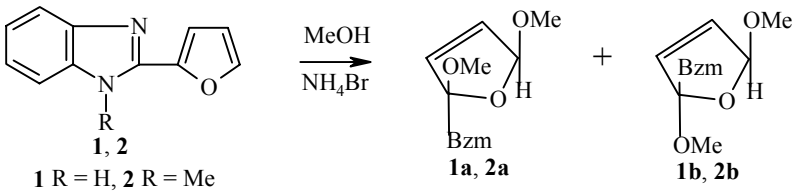
Neither the chemical nor electrochemical methoxylation of furan derivatives of benzimidazole has been studied extensively. We have found that the introduction of a substituent such as 2-benzimidazolyl at $C_{(2)}$ of the furan ring does not hinder the methoxylation reaction as previously observed for bis(aminoalkyl)furans [1].

2-(2'-Furyl)benzimidazole (**1**) and its N-methylation product, 2-(2'-furyl)-1-methylbenzimidazole (**2**) [2] were subjected to electrolysis. The 2',5'-dimethoxy-2',5'-dihydrofuryl derivatives obtained in both cases are ~70:30 mixtures of two geometrical isomers as indicated by ^{1}H NMR spectroscopy [3].



1, **2**

1 R = H, **2** R = Me

1a, **2a**

1b, **2b**

1a, **1b** Bzm = 2-benzimidazolyl

2a, **2b** Bzm = 1-methyl-2-benzimidazolyl

The isomers obtained from **2** were isolated as pure compounds and their structure was established by ^{1}H NMR spectroscopy in $CDCl_3$. Thus, **2a** is likely the *cis* isomer since the singlet for the furan 5'-H proton is downfield relative to the corresponding signal in the spectrum of **2b**. This shift is undoubtedly related to the effect of the benzimidazolyl group. The methoxylation product of **1** could not be separated into isomers.

2-(2',5'-Dimethoxy-2',5'-dihydro-2'-furyl)benzimidazoles 1a and 1b, Isomer Mixture. A sample of **1** (3.68 g, 20 mmol) and ammonium bromide (1.96 g, 20 mmol) were dissolved in methanol (50 ml). The solution was placed in an electrolyzer with a platinum anode and nickel cathode and cooled to -15°C. The temperature was maintained during the experiment from -5 to -15°C. The electrolysis was carried out with anodic current density 0.05 A/cm^2 until $2.90\cdot10^5$-$3.86\cdot10^5$ C/mol (3-4 F/mol) was passed. The reaction mixture was neutralized by adding aqueous ammonia. Methanol was distilled off in vacuum using a water pump. The residue was extracted with CH_2Cl_2, dried over Na_2SO_4, and subjected to chromatography on a 100-cm column

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(d = 2.5 cm) packed with 300 g of alumina (Brockmann grade II activity) using CH_2Cl_2 as the eluent. Yield of the isomer mixture 4.23 g (86%); mp of the picrate 232-233°C. Found, %: C 48.41; H 3.87; N 15.08. $C_{19}H_{17}N_5O_{10}$. Calculated, %: C 48.01; H 3.60; N 14.73.

2-(2',5'-Dimethoxy-2',5'-dihydro-2'-furyl)-1-methylbenzimidazoles (2a) and (2b) were obtained as an isomer mixture by analogy to **1a** and **1b** from **2** (3.96 g, 20 mmol). After chromatography, the following indices were obtained for the individual isomers.

***cis* Isomer 2a** was obtained in 46% yield (1.82 g); mp 64-65°C (pentane). ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm, J (Hz): 3.10 (3H, s, 2'-OMe); 3.28 (3H, s, 5'-OMe); 3.96 (3H, s, N–CH_3); 6.10 (1H, s, 5'-H); 6.25 (1H, d, J = 5.9, 4'-H); 7.15-7.50 (3H, m, Ar); 7.73 (1H, d, J = 8.3, Ar). Found, %: C 65.03; H 6.09; N 11.06. $C_{14}H_{16}N_2O_3$. Calculated, %: C 64.60; H 6.20; N 10.76.

***trans* Isomer 2b** was obtained in 15% yield (0.59 g); mp 68-69°C (heptane). ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm, J (Hz): 3.37 (3H, s, 2'-OMe); 3.53 (3H, s, 5'-OMe); 3.96 (3H, s, N–CH_3); 5.58 (1H, s, 5'-H); 6.25 (1H, d, J = 5.9, 3'-H); 6.58 (1H, d, J = 5.9, 4'-H); 7.15-7.50 (3H, m, Ar); 7.73 (1H, d, J = 8.3, Ar). Found, %: C 64.8; H 5.82. $C_{14}H_{16}N_2O_3$. Calculated, %: C 64.60; H 6.20.

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