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UNEXPECTED CYCLIZATION OF 4'-METHYL-1',4'-DIHYDRO-2,3'-BIQUINOLYL TO BENZO-[5,6]INDOLIZINO[1,2-*c*]QUINOLINE

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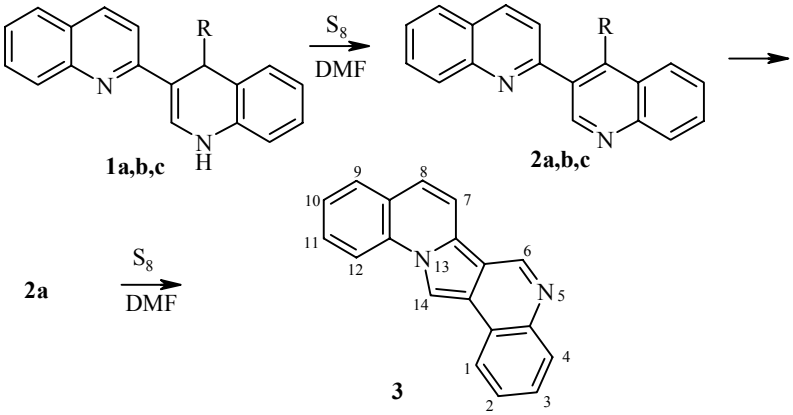
Keywords: benzo[5,6]-indolizino[1,2-*c*]quinoline, 2,3'-biquinolyls, 4'-R-1',4'-dihydro-2,3'-biquinolyls, oxidation, sulfur, cyclization.

Reaction of 4'-methyl-1',4'-dihydro-2,3'-biquinolyl (**1a**) [1, 2] with elemental sulfur in a 1:2.5 mole ratio in boiling DMF for 3 h, followed by purification by column chromatography (silica gel L 40/100, 2-propanol as the eluent) leads to benzo[5,6]indolizino[1,2-*c*]quinoline (**3**) in 27% yield. This reaction probably includes a step involving formation of 4'-methyl-2,3'-biquinolyl (**2a**), since the latter behaves analogously to compound **1** under the above conditions. Furthermore, when the amount of sulfur was decreased two-fold and the reaction time was decreased to 15 min, we could isolate compound **2a** in 44% yield by column chromatography. When dihydrobiquinolyls **1b,c** are used as the substrate, this reaction stops at the step of the corresponding biquinolyls **2b,c** in close to quantitative yield, which also supports the reaction scheme proposed.



1, 2 a R = Me, **b** R = H, **c** R = Ph

Some representatives of the benzo[5,6]indolizino[1,2-*c*]quinoline heterocyclic system were obtained previously from quinolinium salts [3].

4'-Methyl-2,3'-biquinolyl (2a). Mp 137-138°C (benzene–hexane). R_f 0.91 (Silufol UV-254, ethyl acetate). ^{1}H NMR spectrum (200 MHz; $CDCl_3$), δ, ppm, *J*, Hz: 2.80 (3H, s, Me); 7.62 (1H, dd, $J_{6'7'}$ = 6.92, $J_{7'8'}$ = 8.16, 7'-H); 7.63 (1H, d, J_{34} = 8.53, 3-H); 7.67 (1H, dd, J_{56} = 7.98, J_{67}= 6.89, 6-H); 7.77 (1H, dd,

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$J_{5'6'}$ = 8.01, $J_{6'7'}$ = 6.92, 6'-H); 7.81 (1H, dd, J_{67} = 6.89, J_{78} = 8.18, 7-H); 7.93 (1H, d, $J_{5'6'}$ = 8.01, 5'-H); 8.16 (1H, d, $J_{7'8'}$ = 8.16, 8'-H); 8.17 (1H, d, J_{56} = 7.98, 5-H); 8.21 (1H, d, J_{78} = 8.18, 8-H); 8.32 (1H, d, J_{34} = 8.53, 4-H); 9.05 (1H, s, 2'-H). Found, %: C 84.53; H 5.16; N 10.31. $C_{19}H_{14}N_2$. Calculated, %: C 84.42; H 5.22; N 10.36.

2,3'-Biquinolyl (2b, $C_{18}H_{12}N_2$). Mp 175-176°C (benzene); according to data in [4], mp 175-176°C.

4'-Phenyl-2,3'-biquinolyl (2c, $C_{24}H_{16}N_2$). Mp 133-134°C (benzene); according to data in [5], mp 133-134°C.

Benzo[5,6]indolizino[1,2-*c*]quinoline (3). Mp 253-255°C (alcohol). R_f 0.35 (Silufol UV-254, propanol). ^{1}H NMR spectrum (200 MHz; DMSO-d_6), δ, ppm, *J*, Hz: 7.79 (1H, dd, J_{23} = 7.08, J_{34} = 8.55, 3-H); 7.84 (2H, m, 10-, 11-H); 7.99 (1H, dt, J_{12} = 8.8, J_{23} = 7.08, J_{24} = 0.98, 2-H); 8.05 (1H, d, J_{910} = 8.25, 9-H); 8.13 (1H, d, J_{78} = 9.27, 8-H); 8.21 (1H, dd, J_{34} = 8.55, J_{24} = 0.98, 4-H); 8.54 (1H, dd, J_{1112} = 4.95, J_{1012} = 2.2, 12-H); 8.59 (1H, d, J_{78} = 9.27, 7-H); 8.76 (1H, d, J_{12} = 8.8, 1-H); 9.69 (1H, s, 14-H); 9.99 (1H, s, 6-H). Mass spectrum: *m*/*z* (*I*, %) M^+, 268 (100). Found, %: C 85.14; H 4.46; N 10.40. $C_{19}H_{12}N_2$. Calculated, %: C 85.05; H 4.51; N 10.44.

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