

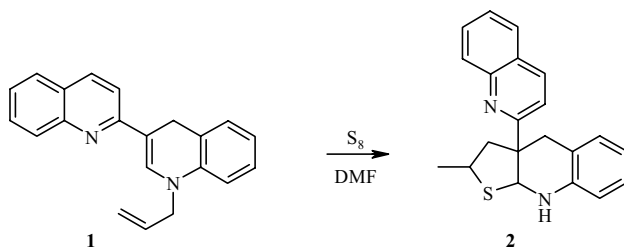
AN UNEXPECTED REACTION OF 1'-ALLYL- 1',4'-DIHYDRO-2,3'-BIQUINOLYL WITH SULFUR

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Keywords: 1',4'-dihydro-2,3'-biquinolyl, sulfur, 3a-(2-quinolyl)-2,3,3a,4,9,9a-hexahydrothieno[2,3-*b*]-quinolines, thiolation.

Previously [1] we have developed a series of methods for the synthesis of 1'-alkyl-1',4'-dihydro-2,3'-biquinolyls which permitted us to study their properties. In this note we report the reaction of 1'-allyl-1',4'-dihydro-2,3'-biquinolyl (**1**) with elemental sulfur.

The reaction of 6 mmol of compound **1** with 12 mmol of elemental sulfur in boiling DMF for 3 h with subsequent extraction with benzene (3 × 50 ml) and column chromatography (silica gel L 40/100, eluent benzene–petroleum ether, 10:1) gave 2-methyl-3a-(2-quinolyl)-2,3,3a,4,9,9a-hexahydrothieno[2,3-*b*]quinoline (**2**). Investigation of the mechanism of this reaction is currently in progress.



2-Methyl-3a-(2-quinolyl)-2,3,3a,4,9,9a-hexahydrothieno[2,3-*b*]quinoline (2). Yield 0.8 g (40%); mp 106–108°C (ethanol). R_f 0.91 (Silufol UV-254, 10:1 benzene–petroleum ether). IR spectrum (KBr disks), ν , cm^{-1} : 3390 (NH). ^1H NMR spectrum (200 MHz, acetone- d_6), δ , ppm, J (Hz): 1.35 (3H, d, $J = 6.41$, Me); 2.44 (1H, dd, $J_{gem} = 12.80$, $J_{3\text{Ha},2} = 10.29$, 3- CH_aH_b); 2.59 (2H, dd, $J_{gem} = 12.80$, $J_{3\text{Hb},2} = 6.40$, 4- CH_aH_b); 3.32 (2H, dd, $J_{gem} = 16.65$, $J_{4\text{Ha},9a} = 1.1$, 4- H_aH_b); 3.57 (1H, d, $J_{gem} = 16.65$, 4- CH_aH_b); 3.91 (1H, m, 2-H); 5.78 (1H, br. d, $J_{\text{NH},9a} = 4.51$, NH); 6.21 (1H, dd, $J_{\text{NH},9a} = 4.51$, $J_{4\text{Ha},9a} = 1.1$, 9a-H); 6.48 (1H, d, $J_{7,8} = 8.11$, 8-H); 6.56 (1H, dd, $J_{5,6} = 7.97$, $J_{6,7} = 7.31$, 6-H); 6.86 (1H, dd, $J_{6,7} = 7.31$, $J_{7,8} = 8.11$, 7-H); 7.06 (1H, d, $J_{5,6} = 7.97$, 5-H); 7.51 (1H, dd, $J_{5',6'} = 8.09$, $J_{6',7'} = 7.54$, 6'-H); 7.58 (1H, d, $J_{3',4'} = 8.53$, 3'-H); 7.71 (1H, dd, $J_{6',7'} = 7.54$, $J_{7',8'} = 8.39$, 7'-H); 7.84 (1H, d, $J_{5',6'} = 8.09$, 5'-H); 7.98 (1H, d, $J_{7',8'} = 8.39$, 8'-H); 8.15 (1H, d, $J_{3',4'} = 8.53$, 4'-H). Mass spectrum: m/z (70 eV): 390 [M - C_3H_6] (98), 289 (100), 257 (40). Found, %: C 75.98; H 6.01; N 8.32. $\text{C}_{21}\text{H}_{20}\text{N}_2\text{S}$. Calculated, %: C 75.87; H 6.06; N 8.43.

REFERENCES

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