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POLAR CYCLOADDITION OF 8-QUINOLINESULFENYL CHLORIDE TO STYRENE

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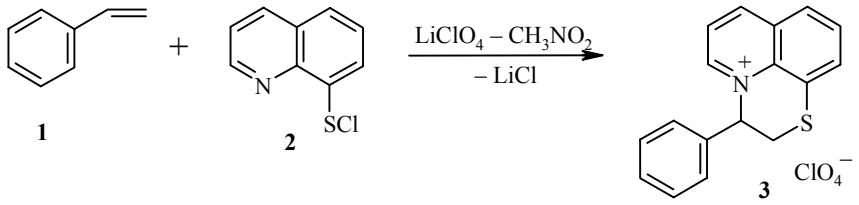
In developing an approach to synthesis of sulfur-containing heterocycles based on ring formation in reactions of sulfenyl chlorides with unsaturated compounds, with ring closure on participation of the nucleophilically active center of the electrophilic reagent [1-3], in this work we have studied the reaction of styrene (**1**) with 8-quinolinesulfenyl chloride (**2**). We have shown that the reaction of these compounds in nitromethane in the presence of lithium perchlorate leads to formation of the *peri*-annelated system **3** in 92% yield: the product of cycloaddition of the sulfur-containing electrophile at the multiple bond.



1 **2** **3**

3-Phenyl-2,3-dihydro[1,4]thiazino[2,3,4-*ij*]quinolinium-4-perchlorate (3). A solution of $LiClO_4$ (1.06 g, 10 mmol) in nitromethane (30 ml) and a solution of sulfenyl chloride **2** (1.96 g, 10 mmol) in nitromethane (15 ml) were added to a solution of alkene **1** (1.04 g, 10 mmol) in nitromethane (10 ml) at 20°C. The mixture was stirred and allowed to stand until the LiCl precipitate fell out of solution. After 30 min, the precipitate was filtered off, the filtrate was evaporated down under vacuum. After recrystallization of the residue from chloroform, we obtained 3.34 g (92%) of compound **3**; mp 203-205°C. IR spectrum (KBr), ν, cm^{-1}: 1652, 1587, 1522, 1493, 1448, 1368, 848 (Het), 1088 (ClO_4). ^{1}H NMR spectrum (DMSO-d_6, 300 MHz), ν, ppm, *J* (Hz): 9.50 d, 9.42 d, 8.33 d, 8.23 t, 8.16 d, 7.96 t (6H, Het); 7.35 d and 6.89 s (5H, Ar); 6.87 (1H, d, ^3J = 3.3, CHN^+); 4.02 dd and 3.93 dd (2H, ^2J = 14.1, CH_2S). ^{13}C NMR spectrum (DMSO-d_6, 50.3 MHz), δ, ppm: 150.67, 150.04, 137.46, 133.45, 133.26, 130.99, 129.19, 128.60, 127.68 (Het), 128.83, 126.28, 125.57, 122.51 (Ar); 68.16 (CHN^+); 29.00 (CH_2S). Found, %: C 56.21; H 3.95; N 3.69; S 8.61. $C_{17}H_{14}ClNO_4S$. Calculated, %: C 56.12; H 3.88; N 3.85; S 8.81. Mass spectrum, *m*/*z* (I_{rel}, %): 264, 263 (M^+ - ClO_4, 43); 232, 230 (57); 188 (12); 161 (100); 77 (55).

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