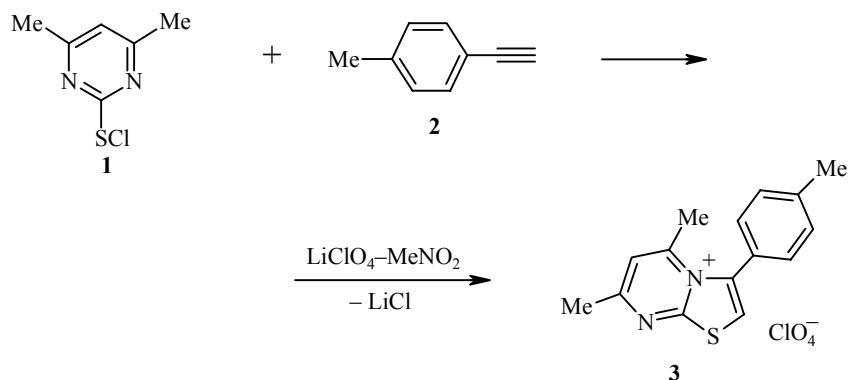


CYCLOADDITION OF 4,6-DIMETHYL-PYRIMIDINE-2-SULFENYL CHLORIDE AT THE ACETYLENE BOND

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We previously described syntheses of sulfur-containing heterocycles based on cycloaddition of hetarene sulfenyl chlorides to alkenes and dienes, with ring closure by means of a nucleophilic active center of the hetaryl moiety [1-5]. In this work, ring formation of the indicated type was studied for the first time using the example of sulfenylation of the carbon – carbon triple bond. We established that reaction of 4,6-dimethylpyrimidine-2-sulfenyl chloride (**1**) with *p*-tolylacetylene (**2**) in nitromethane in the presence of lithium perchlorate occurs as a [3⁺+2] cycloaddition of the sulfenylating reagent at the multiple bond, and leads to the condensed system **3** in 89% yield.



5,7-Dimethyl-3-(4-methylphenyl)thiazolo[3,2-*a*]pyrimidinium Perchlorate (3). A solution of LiClO_4 (1.06 g, 10 mmol) in nitromethane (30 ml) and a solution of sulfenyl chloride **1** (1.75 g, 10 mmol) in nitromethane (15 ml) were added to a solution of arylacetylene **2** (1.16 g, 10 mmol) in nitromethane (15 ml) at 20°C. After 30 min, the LiCl precipitate was filtered out, the filtrate was evaporated down under vacuum. After recrystallization of the residue from methylene chloride, we obtained 3.17 g (89%) of compound **3**; mp 270–272°C (decomposes). IR spectrum (KBr), ν , cm^{-1} : 1648, 1492, 1456, 1408, 1304, 1264, 1140, 986, 756, 728, 702, 620 (Het, Ar), 1088 (ClO_4). ^1H NMR spectrum (DMSO-d_6 , 300 MHz), δ , ppm (J , Hz): 8.25 (1H, s, HCS); 7.86 (1H, s, H_{Het}); 7.54 (2H, d, $^3J = 7.9$, H arom.); 7.39 (2H, d, $^3J = 7.9$, H arom.); 2.78 and 2.44 (6H, s and s, 2CH_3 –Het); 2.23 (3H, s, CH_3 –Ar). Found, %: C 51.02; H 4.17; N 7.78; S 8.92. $\text{C}_{15}\text{H}_{15}\text{ClN}_2\text{O}_4\text{S}$. Calculated, %: C 50.78; H 4.26; N 7.90; S 9.04.

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REFERENCES

1. A. V. Borisov, V. K. Bel'skii, G. N. Borisova, V. K. Osmanov, Zh. V. Matsulevich, and T. V. Goncharova, *Khim. Geterotsikl. Soedin.*, 763 (2001).
2. A. V. Borisov, T. V. Goncharova, G. N. Borisova, V. K. Osmanov, and Zh. V. Matsulevich, *Khim. Geterotsikl. Soedin.*, 407 (2001).
3. A. V. Borisov, T. V. Goncharova, Zh. V. Matsulevich, G. N. Borisova, and V. K. Osmanov, *Khim. Geterotsikl. Soedin.*, 847 (2001).
4. A. V. Borisov, Yu. A. Nikonova, G. N. Borisova, V. K. Osmanov, Zh. V. Matsulevich, and G. V. Zatonskii, *Khim. Geterotsikl. Soedin.*, 855 (2002).
5. A. V. Borisov, T. V. Goncharova, V. K. Osmanov, G. N. Borisova, Zh. V. Matsulevich, N. G. Frolova, and E. D. Savin, *Khim. Geterotsikl. Soedin.*, 1304 (2002).