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SHORT COMMUNICATIONS

New Synthesis of 5-Aryl-5,6-dihydro-7*H*-[1,2,4]triazolo-[5,1-*b*][1,3]thiazin-7-ones

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The acylation of 4,5-dihydro-1,2,4-triazole-5-thiones with carboxylic acid chlorides is known to occur at the nitrogen atom in position *1* of the heteroring [1, 2], while compounds having an activated double bond readily add at the 5-thioxo group at 20°C [3]. Taking these data into account, we proposed a new procedure for synthesizing poorly studied 5-aryl-5,6-dihydro-7*H*-[1,2,4]triazolo[5,1-*b*][1,3]thiazin-7-ones by condensation of triazolethione **I** with substituted cinnamoyl chlorides **IIa**–**IIc** (Scheme 1).

The heterocyclization occurs under mild conditions, on heating the reactants in a benzene–pyridine mixture for 1 h under reflux. Products **III** were formed in good yields, and no by-products were detected.

The structure of compounds **III** was established on the basis on the ^{1}H NMR spectra. The double bond of initial cinnamoyl chloride **II** (two doublets at δ 6.80 and 7.50 ppm) is transformed into a single bond (δ 3.30–5.50 ppm) which gives rise to an *ABX* system. The composition of products **III** was confirmed by the data of mass spectrometry and elemental analysis.

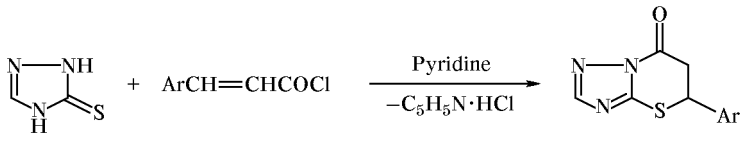
5-Phenyl-5,6-dihydro-7*H*-[1,2,4]triazolo[5,1-*b*]-[1,3]thiazin-7-one (IIIa). A solution of 1.66 g (10 mmol) of cinnamoyl chloride (**IIa**) in 4 ml of benzene was added at 20°C to a solution of 1.01 g (10 mmol) of 4,5-dihydro-1,2,4-triazole-5-thione (**I**) in 4 ml of pyridine. The mixture was refluxed for 1 h, cooled, and diluted with 50 ml of water. The precipitate was filtered off and dried. Yield 1.62 g (70%), mp 163°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 3.47 m (1H, 6-H), 3.88 m (1H, 6-H), 5.40 m (1H, 5-H), 7.46–7.58 m (5H, H_{arom}), 8.10 s (1H, 2-H). Mass spectrum, *m*/*z* (I_{rel}, %): $M^{+\cdot}$ 231 (27). Found, %: C 56.66; H 3.95; N 18.20. $C_{11}H_9N_3OS$. Calculated, %: C 57.14; H 3.89; N 18.18.

Compounds **IIIb** and **IIIc** were synthesized in a similar way.

5-(4-Methoxyphenyl)-5,6-dihydro-7*H*-[1,2,4]triazolo[5,1-*b*][1,3]thiazin-7-one (IIIb). Yield 72%, mp 143°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 3.41 m (1H, 6-H), 3.78 m (1H, 6-H), 3.83 s (3H, CH_3O), 5.36 m (1H, 5-H), 7.00 d (2H, H_{arom}), 7.53 d (2H, H_{arom}), 8.06 s (1H, 2-H). Mass spectrum, *m*/*z* (I_{rel}, %): $M^{+\cdot}$ 261 (41). Found, %: C 55.12; H 4.10; N 16.12. $C_{12}H_{11}N_3O_2S$. Calculated, %: C 55.17; H 4.21; N 16.09.

5-(3-Nitrophenyl)-5,6-dihydro-7*H*-[1,2,4]triazolo[5,1-*b*][1,3]thiazin-7-one (IIIc). Yield 81%, mp 202°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.41 m (1H, 6-H), 3.99 m (1H, 6-H), 5.60 m (1H, 5-H), 7.76 d.d (1H, H_{arom}, J = 8.3, J' = 8.3 Hz), 7.95 d (1H, H_{arom}, J = 8.3 Hz), 8.25 d (1H, H_{arom},

Scheme 1.



I **IIa–IIc** **IIIa–IIIc**

Ar = Ph (**a**), 4-$MeOC_6H_4$ (**b**), 3-$O_2NC_6H_4$ (**c**).

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J = 8.3 Hz), 8.30 s (1H, 2-H), 8.35 s (1H, H_{arom}). Mass spectrum, m/z (I_{rel}, %): $M^{+\cdot}$ 276 (52). Found, %: C 47.85; H 2.90; N 20.60. $C_{11}H_8N_4O_3S$. Calculated, %: C 47.83; H 2.90; N 20.29.

The ^{1}H NMR spectra were recorded on a Varian 300 instrument (300 MHz) using tetramethylsilane as internal reference. The mass spectra were obtained on an MKh-1303 spectrometer.

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