SYNTHESIS OF 3-ALKYL(ARYL)-4,6-DIHYDRO-3H-THIENO[3,4-d]THIAZOLE-2-THIONE 5,5-DIOXIDES

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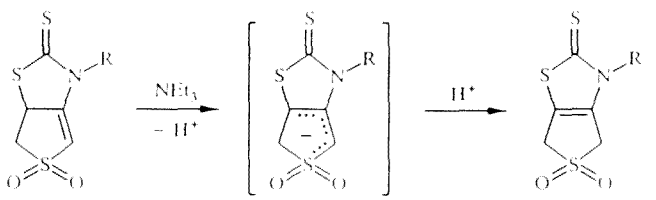
The bicyclic condensed derivatives of 3-thiolene 1,1-dioxide are used as intermediates for the production of analogs of quinodimethanes, which are then introduced as dienes into the Diels–Alder reaction [1, 2]. For the synthesis of such compounds, 3-alkyl(aryl)-4,6-dihydro-3H-thieno[3,4-d]thiazole-2-thione 5,5-dioxides, we proposed to use the isomerization of the double bond by the action of bases in the ring of the previously described 3-alkyl(aryl)-6,7-dihydro-3H-thieno[3,4-d]thiazole-2-thione 5,5-dioxides [3].



Ia, b IIa, b

I, II a R = CH_2Ph, b R = Ph

The transformation is carried out at 50-60°C with 0.005 mole of the corresponding sulfone (Ia, b) and 0.10 g (0.001 mole) of triethylamine in 25 ml of 2-propanol for 1 h. The reaction mixture is cooled, and the precipitate is crystallized from acetonitrile, heated no higher than 60°C. The structure of the obtained compounds was confirmed by the data from the IR and PMR spectra. Thus, the IR spectra contain a weak absorption band at 1640 cm^{-1}, characteristic of the double bond in derivatives of 3-thiolene 1,1-dioxide. In the PMR spectra in the region of 4-5 ppm, there are two singlets for the methylene groups of the dioxothiolene ring.

Compound (IIa). The yield was 93%; mp 158-160°C. IR spectrum (potassium bromide): 1640 (C=C), 1600 (arom), 1325, 1150 (SO_2), 1240 (C=S). PMR spectrum (DMSO-d_6/HMDS): 4.38 (2H, s, CH_2SO_2), 4.40 (2H, s, CH_2SO_2), 5.40 (2H, CH_2N), 7.24-7.34 (5H, m, arom). Found %: S 32.19, N 4.83. $C_{12}H_{11}NO_2S_3$. Calculated %: S 32.34, N 4.71.

Compound (IIb). The yield was 87%; mp 147-149°C. IR spectrum (potassium bromide): 1640 (C=C), 1605 (arom), 1320, 1125 (SO_2), 1260 (C=S). PMR spectrum (DMSO-d_6/HMDS): 4.24 (2H, s, CH_2), 4.48 (2H, s, CH_2), 7.51-7.61 (5H, m, arom). Found %: S 34.22, N 5.04. $C_{11}H_9NO_2S_3$. Calculated %: S 33.94, N 4.94.

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Institute of Bioorganic Chemistry and Petrochemistry, National Academy of Sciences of Ukraine, Kiev. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 12, pp. 1693-1694, December, 1995. Original article submitted May 29, 1995.

0009-3122/95/3112-1471$12.50