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SYNTHESIS OF 2-AROYLMETHYL-5-PHENYL-1,3,4-THIADIAZOLES

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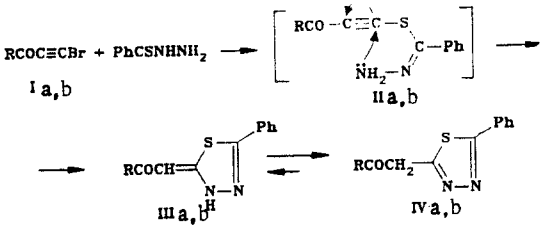
It is known that the reaction of arylthiohydrazides with dimethyl acetylenedicarboxylate and methyl propiolate in refluxing methanol leads to the formation of 5-methoxycarbonylmethyl-5-methoxycarbonyl- and 5-methoxycarbonylmethyl-4,5-dihydro-1,3,4-thiadiazoles [1].

We have established that 2-acylmethyl-5-phenyl-1,3,4-thiadiazoles IVa, b are formed in the reaction of 1-bromo-2-acylacetylenes Ia, b with thiobenzhydrazide in an equimolar ratio in methanol at −30°C in the presence of an equimolar amount of triethylamine. The reaction probably proceeds via a mechanism involving nucleophilic substitution of the bromine atom attached to the ethynyl carbon atom [2, 3] with the formation of intermediate ethynyl sulfides II.



I—IV a R=Ph; b R=2-thienyl

Compound IVa. This compound was obtained in 90% yield and had mp 175-177°C (from MeOH) and M^+ 280. IR spectrum (KBr): 670 (C–S), 1400 (ring C=N), 1550, 1580, 16-5 (aromatic C=C), 1680 cm^{-1} (C=O). ^1H NMR spectrum ($CDCl_3$): 4.88 (2H, s, CH_2), 7.50-8.03 ppm (10H, m, aromatic). ^{13}C NMR spectrum ($CDCl_3$): 193.68 (s, C=O), 170.06, 161.88 [s, $C_{(2)}$ and $C_{(5)}$], 40.00 (s, CH_2), 127.86-134.14 ppm (m, two C_6H_5, eight signals).

Compound IVb. This compound was obtained in 73% yield and had mp 149-151°C (from EtOH) and M^+ 286. IR spectrum (KBr): 695 (C–S), 1410 (ring C=N), 1520, 1580 (aromatic C=C), 1660 cm^{-1} (C=O). ^1H NMR spectrum ($CDCl_3$): 4.80 (2H, s, CH_2), 7.46-7.94 ppm (8H, m, C_6H_5 and C_4H_3S).

The results of elementary analysis of the synthesized compounds corresponded to the calculated values.

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