SYNTHESIS OF Δ^2-1,2,4-TRIAZOLINE DERIVATIVES

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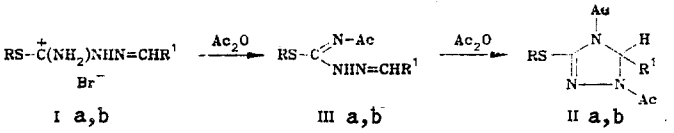
UDC 547.792'236.04

The Δ^2-derivatives are the least studied representatives of the class of 1,2,4-triazolines [1]. It has been shown [2-4] that Δ^2-triazolines can form in the reaction of amidrazones with carbonyl compounds.

We found that thiosemicarbazonium salts I react with acetic anhydride to form 1,4-diacetyl-Δ^2-1,2,4-triazolines (II), the structure of which was proven by x-ray diffraction analysis. The intermediate monoacetyl derivatives III have an acyclic structure [5].



I a,b III a,b II a,b

I—III a,b R=5-nitrofurfuryl-2; a R^1 = furyl-2; b R^1 = Ph

1,4-Diacetyl-3-[(5-nitro-2-furfuryl)thio]-5-(2-furyl))-Δ^2-1,2,4-triazoline (IIa). mp 150-152°C. UV spectrum (EtOH): λ_{max}, nm (log ε): 217 (4.27), 288 (4.15), 314 sh. (4.06). IR spectrum (Nujol): 1697, 1665 cm^{-1}. PMR spectrum ($CDCl_3$): 2.03 (s, 3H, $COCH_3$); 2.20 (s, 3H, $COCH_3$); 4.25 (s, 2H, CH_2S); 7.0 (s, 1H, 5-H); 6.5 and 7.2 – AX-system of β-protons in nitrofuran ring with J_{AX} = 3.7 Hz; 6.35, 6.50 and 7.35 – signals of 4-H, 3-H, and 5-H protons of the furan ring with J_{34} = 3.5; J_{35} = 1.0, and J_{45} = 1.9 Hz. Yield 80%.

1,4-Diacetyl-3-[(5-nitro-2-furfuryl)thio]-5-phenyl-Δ^2-1,2,4-triazoline (IIb). mp 164-165°C. UV spectrum (EtOH): λ_{max}, nm (log ε): 221 (4.29), 294 (4.22), 314 sh. (4.16). IR spectrum (Nujol): 1690, 1665 cm^{-1}. PMR spectrum ($CDCl_3$): 1.86 (s, 3H, $COCH_3$); 2.13 (s, 3H, $COCH_3$), 4.25 (s, 2H, CH_2S); 6.88 (s, 1H, 5-H); 6.53 and 7.18 – AX-system of β-protons in nitrofuran ring with J_{AX} = 3.7 Hz; 7.0-7.5 (m, 5H, C_6H_5). Yield 68%.

The molecular weights (determined mass-spectrometrically) and the elemental analysis data of the compounds obtained correspond to the calculated values.

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Krasnodar Polytechnical Institute, Krasnodar 350670. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 11, pp. 1570-1571, November, 1988. Original article submitted April 6, 1988.

 0009-3122/88/2411-1308$12.50