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BRIEF COMMUNICATIONS

ON THE INTERACTION OF 1-PHENYL-3-CHLORO-2-PYRAZOLINE WITH DIALKYL SULFIDES

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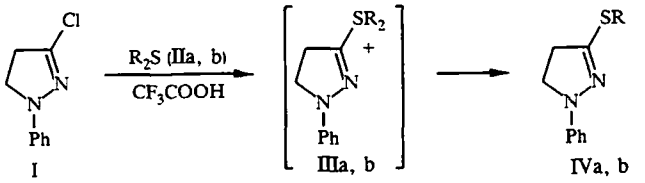
We showed that the reaction of 1-phenyl-3-chloro-2-pyrazoline (I) with the dialkyl sulfides (IIa,b) in the three-molar amount of trifluoroacetic acid leads to the formation of the 1-phenyl-3-alkylthio-2-pyrazolines (IVa,b) with the isolation, in the case of compound (IVa), of butyl chloride and butyl trifluoroacetate (GLC). Thus, the mixture of 1.62 g (0.01 mole) of the chloropyrazoline (I) in 1.5-2 ml of dibutyl sulfide and 3.54 g (0.03 mole) of trifluoroacetic acid is boiled for 2 h. The mixture is concentrated by heating and chromatographed on a dry column of SiO_2 in the 2:1 system of benzene–petroleum ether. The yield of 1-phenyl-3-butylthio-2-pyrazoline (IVa) is 30%, and the mp is 93-95°C. The IR spectrum is characterized at 1610 cm^{-1} (C=N). The PMR spectrum ($CDCl_3$) is as follows: 0.84 ppm, 1.11 ppm, 1.34 ppm, 1.49 ppm (9H, Bu), 3.00 ppm (2H, t, 4-H), 3.50 ppm (2H, t, 5-H), and 6.8-7.4 ppm (5H, m, Ar). Found: M^+ 234, $C_{13}H_{18}N_2S$. Calculated: M 234 (method A).

It can be assumed that the reaction includes the intermediate formation of the salt (III).



II–IV a R = Bu, b R = Me

We also synthesized a compound, identical to the sulfide (IVa) on the basis of PMR and IR, by the reaction of the chloropyrazoline (I) with butyl mercaptan in the presence of triethylamine (method B): the 1:2:2 mixture of the chloropyrazoline (I), triethylamine, and butyl mercaptan in CH_2Cl_2 is maintained for 12 h at 20°C prior to the concentration and isolation by analogy with the method A. The yield of compound (IVa) is 25%, and the mp is 93-95°C.

1-Phenyl-3-methylthio-2-pyrazoline (IVb). This compound is obtained analogously to compound (IVa) by the method A. The yield is 35%, and the mp is 72-74°C. The IR spectrum is characterized at 1610 cm^{-1} (C=N). The PMR spectrum ($CDCl_3$) is as follows: 2.60 ppm (3H, s, CH_3), 3.10 ppm (2H, t, 4-H), 3.80 ppm (2H, t, 5-H), and 6.8-7.5 ppm (5H, m, Ar). Found, %: C 62.36, H 6.49, and N 14.34. $C_{10}H_{12}N_2S$. Calculated, %: C 62.50, H 6.25, and N 14.58.

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