REACTION OF 3-PHENYL-4-DIAZOACETYLSYDNONE WITH DERIVATIVES OF α,β-UNSATURATED ACIDS

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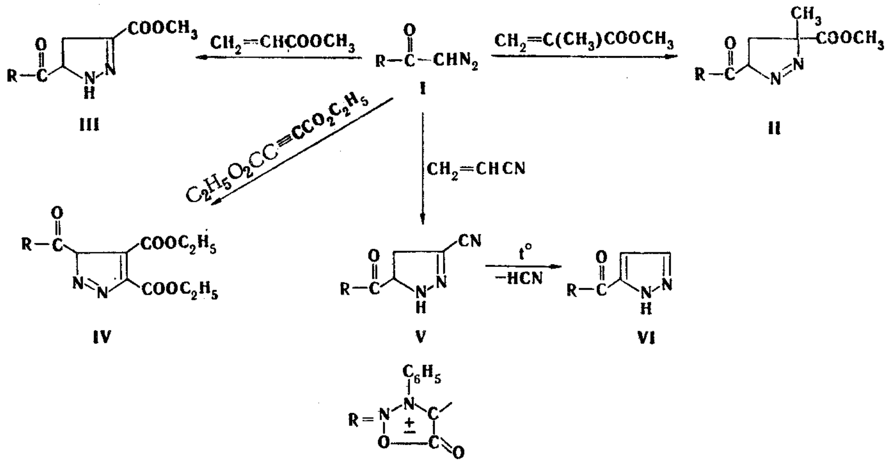
We found that the reaction of 3-phenyl-4-diazoacetylsydnone (I) with methyl esters of acrylic, methacrylic acids or diethyl ester of acetylenedicarboxylic acid in benzene at 50-60°C for 10-12 h proceeds with the retention of the sydnone ring, which usually decomposes under more rigid condtiions, and leads to 1- and 2- pyrazolines (II, III) or pyrazolenine (IV), respectively. The latter compound is not converted into pyrazole under these conditions. It is clear that the formation of enine IV is energetically favorable, because or the enolization of the ketonic carbonyl. In the case of acrylonitrile, 2-pyrazoline (V) forms at 20-25°C, and pyrazole (VI) at 50-60°C, with splitting of the CN group.



The following compounds were obtained after recrystallization from ethanol: II, yield 50%, mp 134-135°C (dec.); λ_{max} (log ε): 288 (3.79), 3.69 nm (4.35); III, yield 51%, mp 156-157°C (dec.); λ_{max} 268 (3.93), 360 nm (4.12); PMR spectrum (in $CHCl_3$): 3.14 (CH_2); 3.70 (OCH_3), 4.57 (CH), 7.42 ppm (Ar); IV, yield 41%, mp 165-167°C (dec.); λ_{max} 255 (3.56), 337 nm (4.18); PMR spectrum (in $CHCl_3$): 1.23 (CH_3); 4.3 (CH_2); 6.8 (CH), 7.5 ppm (Ar); V, yield 32%, mp 123-124°(dec.); VI, yield 41%, mp 159-160°C, λ_{max} 273 (3.78), 357 nm (3.87). IR spectrum: 1785-1765 (C=O of sydnone in II-VI), 1670-1590 (C=O of the keto group in II, III, V, and VI), 3360-3305 (NH in III, V and VI); 1730-1715 (COO in II-IV); 2255 cm^{-1} (C≡N in IV).

The data of elementary analysis for C, H, N correspond to the calculated values.

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*Deceased.

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