INTRAMOLECULAR CYCLIZATION OF ACETYLENIC DERIVATIVES OF AROMATIC CARBOXYLIC ACID HYDRAZIDES

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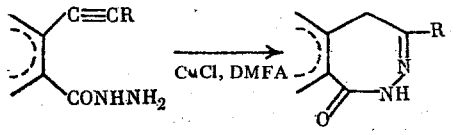
In the presence of Cu(I) salts, aromatic o-acetylenyl-substituted carboxylic acids and amines undergo intramolecular heterocyclization [1, 2]. We found that hydrazides of analogous acetylenic derivatives of carboxylic acids react under the same conditions with closing of a diazepine ring



By heating the hydrazide of 4-phenylethynyl-1-methylpyrazole-5-carboxylic acid [mp 142-143°C (from EtOH). IR spectrum ($CHCl_3$, ν, cm^{-1}): 1675, 1625, 3420 sh, 3350 ($CONHNH_2$), 2225 (C≡C). PMR spectrum ($CDCl_3$, δ, ppm): 3.98 (NH_2), 4.20 (NCH_3), 7.1-7.5 m (C_6H_5, 3-H), 8.38 (NH) in DMFA in the presence of CuCl at 135°C for 30 min, 60% of 7,8-dihydro-1-methyl-5-phenyl-4H-pyrazolo [3,4-d]6,7-diazepin-8-one, mp 197-198°C (from EtOH) were obtained. IR spectrum ($CHCl_3$, ν, cm^{-1}): 1675 (C=O, C=N), 3410 (NH). PMR spectrum ($CDCl_3$, δ, ppm): 4.15 (CH_2), 4.35 (NCH_3), 7.27 (C_6H_5), 7.60 (3-H), 11.17 (NH). 1,2-Dihydro-4-phenyl-5H-benzo[d]-2,3-diazepin-1-one, mp 197-198°C (from EtOH) was synthesized in a similar way. IR spectrum ($CHCl_3$, ν, cm^{-1}): 1680 (C=O), 3415 (NH). PMR spectrum ($CDCl_3$, δ, ppm): 4.29 (CH_2), 7.26 (C_6H_5), 7.70 (6,7,8-H), 8.44 br (9-H), 11.35 (NH). The structure of compounds obtained is also confirmed by elemental analysis.

LITERATURE CITED

1. C. E. Castro, R. Havlin, V. K. Honvald, A. Malte, and S. Moje, J. Am. Chem. Soc., 91, 6464 (1969).
2. S. M. Shvartsberg, S. F. Vasilevskii, T. V. Anisimova, V. A. Gerasimov, Izv. Akad. Nauk SSSR, Ser. Khim., 1342 (1981).

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