SYNTHESIS OF 2-(DIAZOMETHYL)-3-METHYL-5H-FURAN-4-ONE FROM 1,5-BIS(DIAZO)-3-METHYLPENTANE-2,4-DIONE

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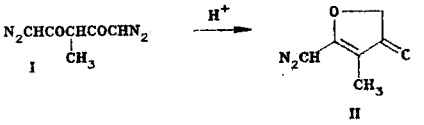
In an investigation of acid reactions of bis(diazoacetyl)alkanes, we observed that unlike the known representatives of the $N_2CHCO(CH_2)nCOCHN_2$ series, where $n \neq 1$ [1], in the presence of acid agents (hydrogen chloride, acetic acid, and silica gel) diazo ketone I undergoes intramolecular cyclization at the oxygen atom of one of the diazocarbonyl groups with the formation of diazomethylfuranone II.



I II

1,5-Bis(diazo)-3-methylpentane-2,4-dione (I) was obtained by the reaction of methylmalonic dichloride with diazomethane in 50% yield, mp 61-62°C (from an ether–hexane mixture). Proton NMR spectrum ($CDCl_3$), δ: 1.37 (3H, doublet, j = 7 Hz, CH_3), 3.37 (1H, quartet, $CH-CH_3$), 5.49 ppm (2H, singlet, CH_2), IR spectrum, 2090 (N≡N), 1600 cm^{-1} (C=O). Uv spectrum (ethanol): λ_{max} 277 nm (log ε 4.25).

2-(Diazomethyl)-3-methyl-5H-furan-4-one (II) was obtained from diazo ketone I by passing I through a column with silica gel L40/100μ in the benzene–ethyl acetate system. Melting point 90-91°C (with decomposition, from ether). Proton NMR spectrum ($CDCl_3$), δ: 1.62 (3H, singlet, CH_2), 4.51 (2H, singlet, (CH_2), 5.08 ppm (1H, singlet, CH). IR spectrum (mineral oil), ν: 2080 (N≡N), 1650 cm^{-1} (C=O). UV spectrum (ethanol): λ_{max} 326 nm (log ε 4.50).

The data of elemental analysis of compounds I and II correspond to the calculated data.

LITERATURE CITED

1. E. Fahr, Ann. Chem., 638, 1 (1960).

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