BROMINATION OF 3-PHENYL-5-(γ-HYDROXYPROPYL)ISOXAZOLE

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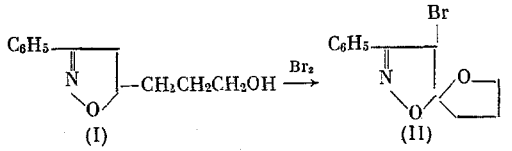
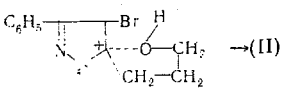
We discovered a new reaction in the series of 5-(γ-hydroxypropyl)isoxazole derivatives (I), and specifically the reaction of (I) with bromine, which in the presence of pyridine leads to the first member of spiro[isoxazoline-5,2'-tetrahydrofuran] (II) in 95% yield. This transformation can be regarded as being



(I) (II)

important proof for the intermediate formation of the σ-complex when an isoxazole is reacted with bromine.



According to [1], an intermediate product of this type is formed in the bromination of a 5-phenacyl-3-phenylisoxazole oxime. Heterocycle (I) was obtained in 75% yield by the reaction of benzonitrile N-oxide with 1-propyn-5-ol as described in [2]. Compound (I), mp 53-55°C (1:1 hexane—benzene). Found: C 71.25; H 6.54; N 6.91%. $C_{12}H_{13}NO_2$. Calculated: C 70.93; H 6.40; N 6.89%. NMR spectrum (δ, ppm, in $CDCl_3$): 6.28 s (CH), 2.04 s (OH), 3.66 t (CH_2O), 2.84 t (CH_2—C=), 1.94 m (CCH_2C). Compound (II), mp 47-48°C (from hexane). Found: C 51.39; H 4.35; N 5.17; Br 28.56%. $C_{12}H_{12}BrNO_2$. Calculated: C 51.06; H 4.26; N 4.96; Br 28.37%. NMR spectrum (δ, ppm, in CCl_4): 4.94 s (CH—Br), 4.60 t (weak splitting, CH_2O), ~2.17 m (2 CH_2).

LITERATURE CITED

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2. H. Kano, I. Adachi, R. Kido, and K. Hirose, J. Med. Chem., 10, 411 (1967).

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