METHOD OF OBTAINING 2-ACETYLPYRROLE-3,4-DICARBOXYLIC ACID

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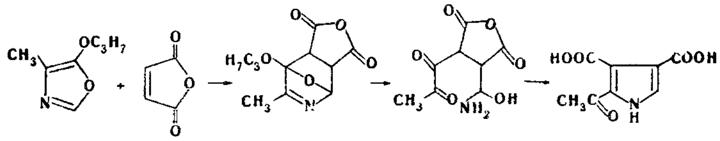
It is known that maleic anhydride is one of the most active dienophiles in heterodiene condensation with 5-alkoxy-4-methyloxazoles, and this is used as a method for synthesizing 3-hydroxy-2-methylcinchomeronic acid and its derivatives [1]. The capacity of the adducts of the heterodiene condensation of 5-ethoxycarbonyloxy-4-methyloxazole with diethyl fumarate on aromatization in an aqueous medium for giving substituted acetylpyrroles has been reported previously [2]. When the aromatization of the adduct (I) was performed under similar conditions, 2-acetylpyrrole-3,4-dicarboxylic acid (II) was formed; it is a lemon-yellow crystalline substance sparingly soluble in water and ether, isomeric with 3-hydroxy-2-methylcinchomeronic acid but possessing different UV and IR spectra. It gives a 2,4-dinitrophenylhydrazone and a Schiff's base with p-phenetidine under standard conditions. The formation of 2-acetylpyrrole-3,4-dicarboxylic acid (II) apparently takes place in the following way:



I

II

2-Acetylpyrrole-3,4-dicarboxylic Acid (II). A solution of 1.96 g of maleic anhydride, 2.82 g of 4-methyl-5-propoxyoxazole, and 40 mg of hydroquinone in 40 ml of ether was stirred at 18-20°C for 1 h 30 min. The reaction mixture was cooled to 5°C and, with stirring, 10 ml of 5% hydrochloric acid was added. The precipitate that deposited was separated off and dried. This gave 1.77 g (45%) of (II). Mp 208-210°C (decomp.). According to the literature [2], mp 210°C (decomp.). UV spectrum, λ_{max}, n (log ε): in 0.1 N NaOH 363 (4.50); in 0.1 N HCl 3.98 (4.20). IR spectrum, cm^{-1}: 3420, 3290, 1750, 1730, 1705, 1665, 1600, 1562, 1510. Found, %: C 48.5; H 3.8; N 7.2. $C_8H_7NO_5$. Calculated, %: C 48.7; H 3.6; N 7.1.

2,4-Dinitrophenylhydrazone: mp 225-226°C (decomp.). UV spectrum in ethanol, λ_{max}, nm (log ε): 432 (4.20). Found, %: C 44.2; H 3.0; N 18.8. $C_{14}H_{11}N_5O_8$. Calculated, %: C 44.6; H 2.9; N 18.6.

Schiff's base with p-phenetidine: mp 186-187°C (decomp.). UV spectrum in ethanol, λ_{max}, nm (log ε): 418 (4.38).

LITERATURE CITED

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2. M. Murakami, K. Takahashi, J. Matsumoto, K. Tamazawa, K. Murase, M. Iwanami, and H. Iwamoto, Bull. Chem. Soc. Japan, 41, 628 (1968).

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