SYNTHESIS OF 3-(1-ADAMANTYL)AMINO-1,2-DIHYRO-5-CHROMENO[4,3-b]PYRAZINE-2,5-DIONE

T. N. Yanborisov, S. N. Shurov, Yu. S. Andreichikov, V. G. Baklykov, and V. L. Savel'ev

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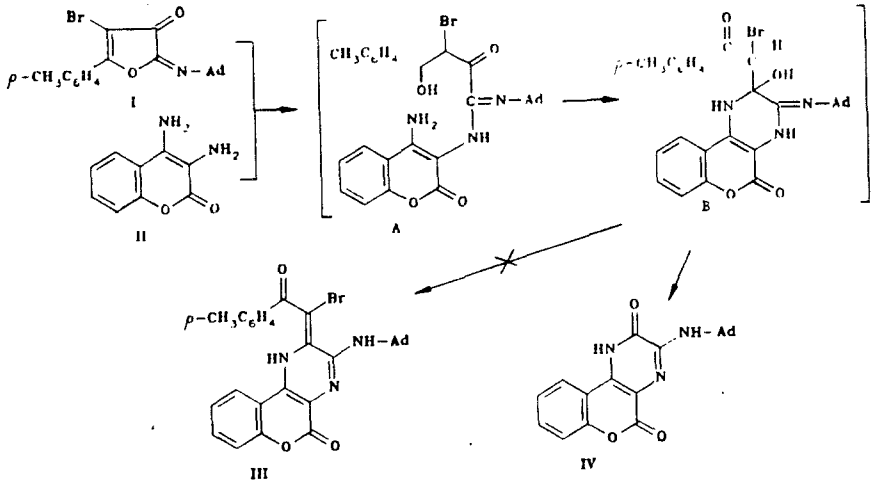
o-Phenylenediamine is known to cleave the ring in N-substituted 2-imino-5-aryl-2,3-dihydro-3-furanones under mild conditions to give 2-N-substituted amino-3-phenacylidene-3,4-dihydroquinoxalines [1].

In a study of the reaction of 2-[N-(1-adamantyl)imino]-4-bromo-5-p-tolyl-2,3-dihydro-3-furanone (I) with 3,4-diaminocoumarin (II), instead of the expected 3-(1-adamantyl)amino-2-(p-toluoyl)bromomethylene-1,2-dihydro-5H-chromeno[4,3-b]pyrazin-5-one (III) there was obtained 3-(1-adamantyl)amino-1,2-dihydro-5H-chromeno[4,3-b]pyrazine-2,5-dione (IV).

The first step appears to involve opening of the furan ring by the more nucleophilic amino-group at $C_{(3)}$ of the 3,4-diaminocoumarin, followed by recyclization of the intermediate amidine A with elimination of ω-bromo-p-methylacetophenone from the intermediate B to give (IV).



I, III, IV, Ad = adamantyl

Compound (IV), $C_{21}H_{21}N_3O_3$. To a solution of 0.4 g (1 mmole) of (I) in 15 ml of dry dioxane was added a solution of 0.17 g (1 mmole) of the diamine (II) in 30 ml of dry dioxane. The mixture was kept at room temperature for 1 h, and the solid which separated was then filtered off and recrystallized from DMF to give 0.32 g (89%) of (IV), mp >350°C. IR spectrum (KBr): 3395 (NH), 1730 ($C_{(5)}O$), 1650 cm^{-1} (C_2O). UV spectrum (in alcohol), λ_{max} (log ε): 348 nm (452). PMR spectrum (DMSO-D_6): 1.65 and 2.05 (15H, two s, adamantyl); 6.31 (1H, br.s, NH); 7.58 ppm (4H, m, aromatic protons). M^+ 363 (by mass spectrometry).

The elemental analysis was in agreement with the calculated values.

LITERATURE CITED

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