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SHORT COMMUNICATIONS

Spiro Heterocyclization of Dimethyl 1-(4-Methylphenyl)-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2,3-dicarboxylate by the Action of 3-Arylamino-5,5-dimethylcyclohex-2-en-1-ones

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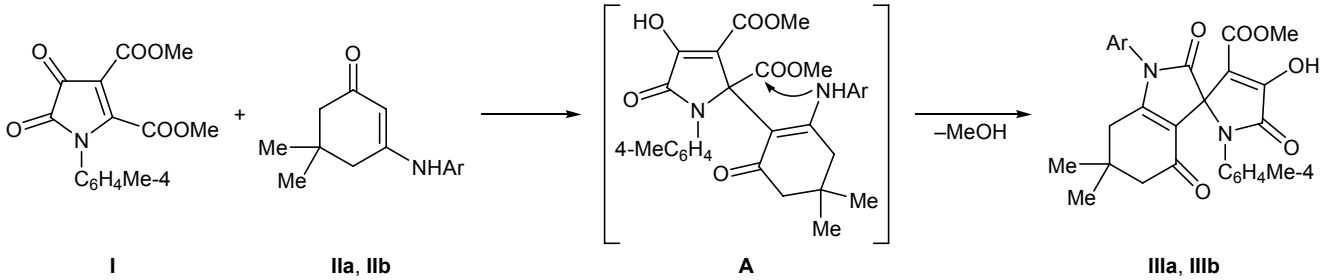
Reactions of dialkyl 4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2,3-dicarboxylates with enamines were reported previously. By reaction of 1-(4-methylphenyl)-4,5-dioxo-4,5-dihydro-1*H*-pyrrole-2,3-dicarboxylate (**I**) with an equimolar amount of 3-arylamino-5,5-dimethylcyclohex-2-en-1-one **IIa** or **IIb** in boiling toluene (reaction time 3–4 h) we obtained methyl 1-aryl-1′-(4-methylphenyl)-4′-hydroxy-6,6-dimethyl-2,4,5′-trioxo-1,1′,2,4,5,5′,6,7-octahydrospiro[indole-3,2′-pyrrole]-3′-carboxylates **IIIa** and **IIIb**. The spectral parameters of compounds **IIIa** and **IIIb** turned out to be quite similar to those of model 3′-benzoyl-1-cyclohexyl-4′-hydroxy-6,6-dimethyl-1′-phenyl-1,1′,2,4,5,5′,6,7-octahydrospiro[indole-3,2′-pyrrole]-2,4,5′-trione whose structure was proved by X-ray analysis [1].

Presumably, compounds **IIIa** and **IIIb** are formed as a result of initial addition of the activated β-CH group in cyclic enamine **II** to the carbon atom in position *2* of pyrroledione **I** with formation of intermediate **A** which undergoes intramolecular cyclization via nucleophilic attack by the NH group on the ester carbonyl carbon atom on C^2 and elimination of methanol.

Methyl 4′-hydroxy-6,6-dimethyl-1′-(4-methylphenyl)-2,4,5′-trioxo-1-phenyl-1,1′,2,4,5,5′,6,7-octahydrospiro[indole-3,2′-pyrrole]-3′-carboxylate (IIIa). A solution of 1.0 mmol of pyrroledione **I** and 1.0 mmol of enamine **IIa** in 20 ml of anhydrous toluene was heated for 4 h (the progress of the reaction was monitored by TLC). The mixture was cooled, and the precipitate was filtered off and recrystallized from toluene. Yield 71%, mp 240–241°C (from toluene). IR spectrum, ν, cm^{-1}: 3170 (OH); 1750, 1701 (C^2=O, $C^{5'}$=O, COOMe); 1628 (C^4=O). ^{1}H NMR spectrum, δ, ppm: 0.55 s and 0.93 s (3H each, Me), 2.00 d (1H, 7-H, J = 16.3 Hz), 2.11 d (1H, 5-H, J = 18.5 Hz), 2.18 d (1H, 7-H, J = 16.3 Hz), 2.30 s (3H, Me), 2.38 d (1H, 5-H, J = 18.5 Hz), 3.70 s (3H, OMe), 6.94–7.60 m (9H, H_{arom}), 12.08 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 20.63 (Me), 26.51 (6-Me), 27.85 (6-Me), 33.82 (C^6), 35.86 (C^7), 50.48 (C^5), 51.56 (OMe), 68.31



I **IIa**, **IIb** **A** **IIIa**, **IIIb**

Ar = Ph (**a**), 4-MeC_6H_4 (**b**).

(C^3), 107.94 (C^{3a}), 109.52 ($C^{3'}$), 125.26–138.03, 155.04 ($C^{5'}$), 161.99 (C^2), 165.04 (COO), 174.45 ($C^{4'}$), 190.56 (C^4). Found, %: C 69.15; H 5.34; N 5.77. $C_{28}H_{26}N_2O_6$. Calculated, %: C 69.12; H 5.39; N 5.76.

Methyl 4′-hydroxy-6,6-dimethyl-1,1′-bis(4-methylphenyl)-2,4,5′-trioxo-1,1′,2,4,5,5′,6,7-octahydrospiro[indole-3,2′-pyrrole]-3′-carboxylate (IIIb) was synthesized in a similar way. Yield 78%, mp 234–236°C (from toluene). IR spectrum, ν, cm^{-1}: 3280 (OH); 1759, 1719 (C^2=O, $C^{5'}$=O, COOMe); 1613 (C^4=O). ^{1}H NMR spectrum, δ, ppm: 0.56 s and 0.93 s (3H each, Me), 2.00 d (1H, 7-H, J = 16.2 Hz), 2.10 d (1H, 5-H, J = 18.0 Hz), 2.17 d (1H, 7-H, J = 16.2 Hz), 2.30 s (3H, Me), 2.34 d (1H, 5-H, J = 18.0 Hz), 2.37 s (3H, Me), 3.70 s (3H, OMe), 6.93–7.37 m (8H, C_6H_4), 12.53 br.s (1H, OH). Found, %: C 69.60; H 5.61; N 5.66. $C_{29}H_{28}N_2O_6$. Calculated, %: C 69.59; H 5.64; N 5.60.

The IR spectra were recorded on an FSM-1201 spectrometer from samples dispersed in mineral oil. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker AM-400 spectrometer at 400 and 100 MHz, respectively, using DMSO-d_6 as solvent and tetramethylsilane as internal reference. The purity of the products was checked by TLC on Silufol plates using ethyl acetate as eluent.

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REFERENCE

1. Bannikova, Yu.N., Maslivets, A.N., and Aliev, Z.G., *Russ. J. Org. Chem*., 2004, vol. 40, p. 1791.