

SHORT
COMMUNICATIONS

Direct Heterocyclization of Tetrahydroisoquinolin-1-ylideneacetic Acids by the Action of 5-Arylfuran-2,3-diones

V. V. Khalturina^a, Yu. V. Shklyayev^a, Z. G. Aliev^b, and A. N. Maslivets^c^a Institute of Technical Chemistry, Ural Division, Russian Academy of Sciences, Perm, Russia^b Institute of Chemical Physics Problems, Russian Academy of Sciences, Chernogolovka, Moscow oblast, Russia^c Perm State University, ul. Bukireva 15, Perm, 614990 Russia
e-mail: koh2@psu.ru

Received April 2, 2009

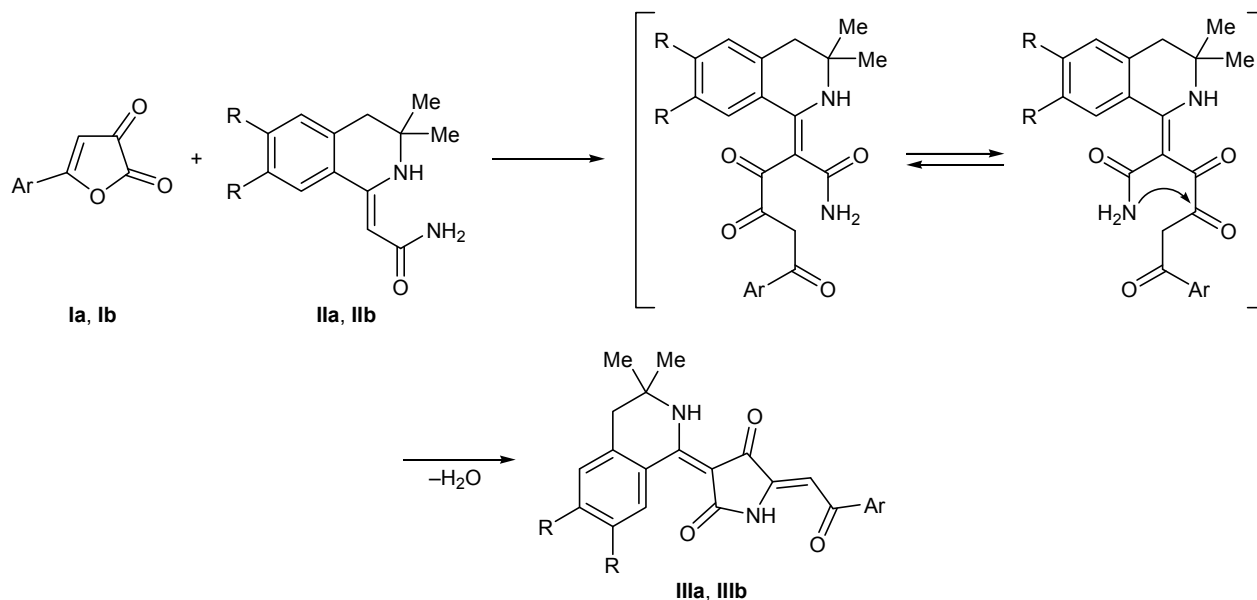
DOI: 10.1134/S1070428009100297

We previously described acylation with 5-arylfuran-2,3-diones of the β -CH group in the enamino tautomers of substituted 1-methyl-3,4-dihydroisoquinolines having no other functional groups. These reactions afforded (2*Z*,5*Z*)-1-aryl-3-hydroxy-5-[3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]pent-2-ene-1,4-diones [1, 2]. In the present communication we report on the reaction of 5-arylfuran-2,3-diones with 1-methylidene-1,2,3,4-tetrahydroisoquinolines possessing an additional functional group at the exocyclic carbon atom attached to C¹.

5-Arylfuran-2,3-diones **Ia** and **Ib** reacted with [3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]-

acetamides **IIa** and **IIb** at a ratio of 1:1 on heating in boiling anhydrous benzene (reaction time 1–2 h). The progress of the reactions was monitored by thin-layer chromatography, and the products were the corresponding (3*E*,5*Z*)-5-(2-aryl-2-oxoethylidene)-3-[3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]pyrrolidine-2,4-diones **IIIa** and **IIIb** whose structure was determined by X-ray analysis.

Presumably, the first reaction step is acylation of the β -CH group in the enamino fragment of isoquinolines **IIa** and **IIb** by the C²=O carbonyl carbon atom of 5-arylfuran-2,3-diones **Ia** and **Ib**. The subsequent opening of the furan ring at the O¹–C² bond (as report-



I, Ar = Ph (**a**), 4-MeC₆H₄ (**b**); **II**, R = H (**a**), MeO (**b**); **III**, Ar = Ph, R = H (**a**), Ar = 4-MeC₆H₄, R = MeO (**b**).

ed for the reactions of 5-arylfuran-2,3-diones with oxygen-, sulfur-, and nitrogen-centered mono- and binucleophiles [3]) is followed by *Z,E* isomerization and intramolecular cyclization with elimination of water molecule.

(3*E*,5*Z*)-3-[3,3-Dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]-5-(2-oxo-2-phenylethylidene)pyrrolidine-2,4-dione (IIIa). A solution of 2.0 mmol of 5-phenylfuran-2,3-dione (**Ia**) and 2.0 mmol of amide **IIa** in 20 ml of anhydrous benzene was heated for 2 h under reflux. The mixture was cooled, and the precipitate was filtered off. Yield 0.60 g (85%), mp 166–168°C (decomp., from EtOH). IR spectrum, ν , cm^{-1} : 3383, 3190 br (NH), 1661 ($\text{C}^2=\text{O}$), 1625 ($\text{C}^4=\text{O}$), 1594 (PhCO). ^1H NMR spectrum, δ , ppm: 1.29 s (6H, Me), 3.00 s (2H, 4-H), 6.69 s (1H, CH), 7.37–8.30 m (9H, H_{arom}), 10.37 s (1H, NH), 11.25 s (1H, 1-H). Found, %: C 74.03; H 5.53; N 7.33. $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_3$. Calculated, %: C 74.18; H 5.41; N 7.52.

(3*E*,5*Z*)-3-[6,7-Dimethoxy-3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]-5-[2-(4-methylphenyl)-2-oxoethylidene]pyrrolidine-2,4-dione (IIIb) was synthesized in a similar way. Yield 87%, mp 111–113°C (decomp., from EtOH). IR spectrum, ν , cm^{-1} : 3336, 3180 br (NH), 1678 ($\text{C}^2=\text{O}$), 1633 ($\text{C}^4=\text{O}$), 1611 ($=\text{CHC}=\text{O}$). ^1H NMR spectrum, δ , ppm: 1.30 s (6H, Me), 2.35 s (3H, MeC_6H_4), 2.93 s (2H, 4-H), 3.82 s (3H, OMe), 3.89 s (3H, OMe), 6.67 s (1H, CH), 6.99–

8.12 m (6H, H_{arom}), 10.26 s (1H, NH), 11.25 s (1H, 1-H). Found, %: C 69.93; H 6.07; N 6.09. $\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_5$. Calculated, %: C 69.94; H 5.87; N 6.27.

The IR spectra were recorded on an FMS-1201 spectrometer from samples dispersed in mineral oil. The ^1H NMR spectra were measured on a Bruker WP-400 instrument from solutions in $\text{DMSO}-d_6$ using TMS as internal reference. The purity of the isolated compounds was checked by thin-layer chromatography on Silufol plates using ethyl acetate–benzene (1:5) as eluent; development with iodine vapor.

This study was performed under financial support by the Russian Foundation for Basic Research (project nos. 08-03-01032, 07-03-96036, 07-03-00001).

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