

LETTERS
TO THE EDITOR

The Interaction of Thiazolidine-2,4-dione with an Aromatic Aldehyde Under Microwave Irradiation

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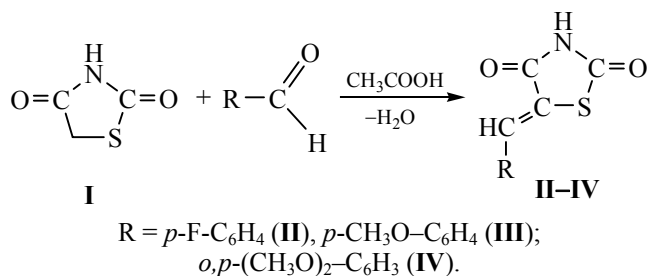
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Due to the development and use of the resource-saving technologies, the effect of microwave field on the known organic reactions has been comprehensively investigated [1–3]. The advantages of microwave irradiation over traditional activation sources are uniform heating of the reaction mixture, heating zero lag, and significant reduction of the reaction time.

We explored the possibility of thiazolidine-2,4-dione 5-arylidene derivatives **II–IV** synthesis under microwave irradiation according to the following scheme.



Thiazolidine-2,4-dione derivatives were prepared under standard conditions with convection heating (70–80°C, 8–9 h). Then the reaction mixture was concentrated, and the desired product was isolated [3, 4]. The main drawback of the method was the reaction duration.

In the case of microwave activation, the reaction conditions were investigated by changing the irradiation duration (1 to 20 min) and irradiation power (70 to 750 W). The optimal conditions were: irradiation power of 500 W and reaction duration of 2–5 min. Under those conditions, yield of **II–IV** was of 65–72%.

The so prepared thiazolidine-2,4-dione derivatives were yellow (**II**) or white (**III**, **IV**) crystalline substances, soluble in polar organic solvents. Structure and purity of the obtained compounds were confirmed by ¹H NMR spectroscopy and elemental analysis.

General procedure for the synthesis of thiazolidine-2,4-dione derivatives II–IV under convection heating. 1.64 g (0.02 mol) of anhydrous sodium acetate and 2.65 g (0.025 mol) of an aromatic aldehyde in 10 ml of acetic acid was added to 2.34 g (0.02 mol) of thiazolidine-2,4-dione **I**. The reaction mixture was refluxed for 8–9 h. The course of the reaction was monitored by TLC. The resulting precipitate was filtered off and recrystallized from acetic acid.

General procedure for the synthesis of thiazolidine-2,4-dione derivatives II–IV under microwave activation. 1.64 g (0.02 mol) of anhydrous sodium acetate and 2.65 g (0.025 mol) of an aromatic aldehyde in 10 ml of acetic acid was added to 2.34 g (0.02 mol) of thiazolidine-2,4-dione **I**. The reaction mixture was irradiated for 5 min at 500 W with short interruptions of irradiation every 10 s. The reaction progress was monitored by TLC. After cooling, the formed precipitate was filtered off and recrystallized from acetic acid.

5-(4-Fluorobenzylidene)thiazolidine-2,4-dione (II). Yield 1.97 g (48%) (convection heating), 3.5 g (78%) (microwave activation), yellow powder, mp 218–219°C. ¹H NMR spectrum (500 MHz), δ, ppm (*J*, Hz): 7.39 d (2H, Ar, *J* 8.9), 7.67 d (2H, Ar, *J* 8.6), 7.81 s (1H, CH=), 12.55 s (1H, NH).

5-(4-Methoxybenzylidene)thiazolidine-2,4-dione (III). Yield 1.77 g (43%) (convection heating), 3.1 g (67%) (microwave activation), white powder, mp 208–210°C. ^1H NMR spectrum (500 MHz), δ , ppm (J , Hz): 3.82 s (3H, OCH_3), 7.11 d (2H, Ar, J 8.8), 7.55 d (2H, Ar, J 8.8), 7.56 s (1H, $\text{CH}=\text{}$), 12.51 s (1H, NH).

5-(2,4-Dimethoxybenzylidene)thiazolidine-2,4-dione (IV). Yield 1.4 g (34.4%) (convection heating), 3.71 g (70%) (microwave activation), white powder, mp 240–242°C. ^1H NMR spectrum (500 MHz), δ , ppm (J , Hz): 3.85 s (3H, OCH_3), 3.89 s (3H, OCH_3), 6.68 d (2H, Ar, J 2.37), 6.70 d.d (2H, Ar, J 2.40), 7.34 d (1H, Ar, J 8.58), 7.9 s (1H, $\text{CH}=\text{}$), 12.45 s (1H, NH).

The ^1H NMR spectra were recorded with Bruker DRX500 spectrometer (500 MHz) in $\text{DMSO}-d_6$ with TMS as internal standard. TLC analysis was performed on Sorbfil plates using isopropyl alcohol–benzene–

ammonia mixture (10:5:2) as eluent and detection with iodine vapors. Melting points were determined using Boetius apparatus.

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