Synthesis of a 2-Furylpyrazoline Derivative Using Microwave Irradiation

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Abstract: A simple method for the synthesis of pyrazoline derivative containing furan moiety was developed. Thus, 5-(6-bromo-1,3-benzodioxol-5-yl)-3-(2-furyl)-1-(3-methylphenyl)-4,5-dihydro-1H-pyrazole was synthesized using microwave irradiation and it was characterized by NMR, IR, and LCMS.

Keywords: (2Z)-3-(6-bromo-1,3-benzodioxol-5-yl)-1-(2-furyl) prop-2-en-1-one; 3-tolylhydrazine hydrochloride; microwave

Aryl furylpyrazolines exhibit antidepressant and anticonvulsant activities [1]. Pyrazolines can be synthesized by the reaction between chalcones (1) and arylhydrazines (2) using catalytic amounts of acetic acid in ethanol as solvent under reflux conditions [2] and acetic acid as a solvent [3]. In some cases bases like K2CO3 [4] and Ba(OH)2 [5] were employed. We are hereby reporting a simple method for synthesizing an aryl furylpyrazoline by a non-conventional method, using microwave conditions, which does not need any catalyst. The work-up procedure is simple and convenient.
A solution of (1) (0.321 g, 1 mmol) which was prepared by the reaction between 6-Bromopiperonal and 2-Acetylfuran and (2) (0.158g,1mmol) in absolute ethanol (5 ml) was placed in a microwave Pyrex vial and irradiated with 200W for 10 min at 150 °C (final temperature). The reaction mixture was cooled to room temperature and concentrated. The solid obtained was washed with a little amount of hexane, filtered and dried under vacuum to give a brown-coloured solid (3).

Yield = 80%

M.p. = 138 °C

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.53$ (d, $J = 1.6$ Hz, 1H), 7.10 (t, $J = 7.6$ Hz, 2H), 7.03 (s, 1H), 6.71 (s, 1H), 6.66 (t, $J = 7.6$ Hz, 2H), 6.59 (d, $J = 3.6$ Hz, 1H), 6.50-6.49 (m, 1H), 5.93 (s, 2H), 5.53 (q, $J = 10.6$ Hz, 1H), 3.91-3.84 (m, 1H), 2.95 (dd, $J = 6.8$ Hz, 17.6 Hz, 1H), 2.33 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): 148.22, 148.03, 147.92, 143.95, 143.51, 139.05, 138.98, 134.14, 128.86, 120.48, 114.15, 112.90, 111.73, 111.68, 110.17, 109.79, 107.33, 101.86, 63.01, 41.87 and 21.74.

MS: m/z (ES), 426 [(M+1)$^-$].

IR: cm$^{-1}$ = 3226, 2903, 2359, 1598, 1582, 1409, 1364, 1318, 1240, 1159, 1109, 1035, 1006, 932, 870, 852, 752, 690, 668, 596, 538.

Elemental Analysis: Calculated for C$_{21}$H$_{17}$BrN$_2$O$_3$ (425.27): C, 59.3%; H, 4.03%; N, 6.59%.
Found: C, 58.05%; H, 4.18%; N, 6.26%.

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References and Notes


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