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4-[5-(4-methylphenyl)-2-furfuryl]iden-2-butenolide

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The general part of the experimental section [1] has been presented elsewhere. 2-Butenolide (7.6 ml, 0.1 mol) and piperidine (2.5 ml, 0.025 mol) were dissolved in ethanol (50 ml), and then under stirring a solution of 5-(4-methyl-phenyl)furfural (9.3 g, 0.05 mol) in 50 ml of ethanol was added to this mixture. The reaction mixture was left to stand at the room temperature for three days until the initial aldehyde was consumed (control TLC). After that the reaction mixture was cooled to crystallize the desired product. The obtained crystals were filtered off and recrystallized from ethanol giving 11.3 g (90 %) of 4-[5-(4-methylphenyl)-2-furfuryl]iden-2-butenolide.

M.p. 147-148°C (ethanol).

UV (EtOH) $[l_{max} (nm), log e (dm^3 mol^{-1} cm^{-1})]$: 275 (3.92), 420 (4.12).

IR (cm⁻¹): 1640 (C=C); 1750 (C=O), 1780 (C=O).

¹H NMR (CDCl₃, 250 MHz): 7.65 (d, 2H, o-H_{Ar}, J = 8.4 Hz); 7.60 (d, 2H, m-H_{Ar}, J = 8.4 Hz); 7.49 (d, 1H, H_B, J_{BA} = 5.2 Hz); 7.15 (d, 1H, H_D, J_{DE} = 3.5 Hz); 6.78 (d, 1H, H_E, J_{ED} = 3.5 Hz); 6.18 (dd, 1H, H_A, J_{AB} = 5.2 Hz, J_{AC} = 0.8 Hz); 6.13 (br s, 1H, H_C, J_{CA} = 0.8 Hz); 2.38 (s, 3H, CH₃).

EI-MS: 252 (M⁺ 100%); 224 (17%).

Anal. calc. for $C_{16}H_{12}O_3$ (252.26): C 76.15, H 4.80; Found C 75.89, H 4.70.

Reference

1. Sorotskaya, L.N.; Badovskaya, L.A.; Kaklyugina, T.Y.; Belen'kij, L.I.; Ignatenko, A.V.; Krutoshikova, A.; Panieva, L.A. *Zhurnal Organich. Khimii (Journal of Organic Chemistry - Russian Edition)* **1989**, *25*, 175.

Sample availability: available from authors (Dr. Lyudmila N. Sorotskaya) and MDPI.

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