

Short Note

2*E*,2'*E*-3,3'-(1,4-Phenylene)bis(1-(2,5-dimethylfuran-3-yl)prop-2-en-1-one

Abdullah M. Asiri ^{1,2,*} and Salman A. Khan ¹

¹ Chemistry Department, Faculty of Science, King Abdul Aziz University, P.O. Box 80203, Jeddah, Saudi Arabia

² The Center of Excellence for Advanced Materials Research, King Abdul Aziz University, Jeddah, P.O. Box 80203, Saudi Arabia

* Author to whom correspondence should be addressed; E-Mail: aasiri2@kau.edu.sa; Tel.: +966 2 6952293; Fax: +966 2 6952292.

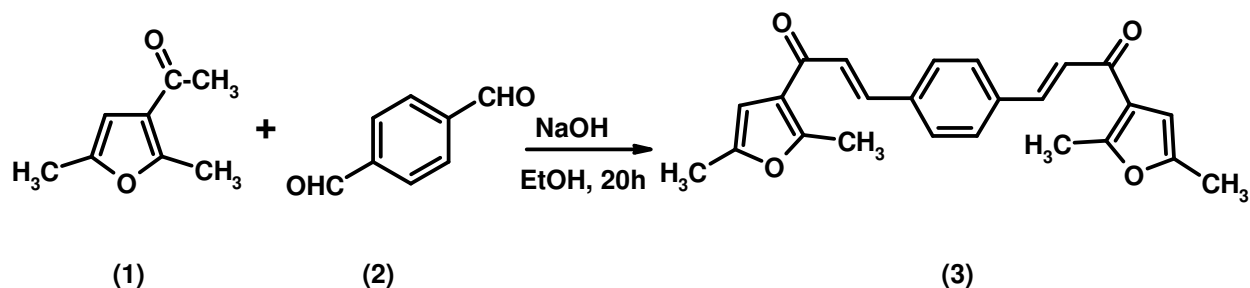
Received: 8 July 2010 / Accepted: 14 September 2010 / Published: 15 September 2010

Abstract: A bis-chalcone has been synthesized by reaction of 3-acetyl-2,5-dimethylfuran and terephthalaldehyde in ethanolic NaOH at room temperature: (2*E*,2'*E*)-3,3'-(1,4-phenylene)bis(1-(2,5-dimethylfuran-3-yl)prop-2-en-1-one) (**3**) was obtained in high yield. The structure of this compound was established by elemental analysis, IR, ¹H NMR, ¹³C NMR and EI-MS spectral analysis.

Keywords: chalcone; terephthalaldehyde; 3-acetyl-2,5-dimethylfuran

The Claisen-Schmidt condensation is the most important reaction for the formation of 1,3-diphenyl-2-propene-1-ones. The products, also known as chalcones, generally are synthesized by this method from suitable acetophenones and benzaldehydes. Chalcones are considered to be precursors of flavonoids when found as naturally-occurring compounds. The chemical importance of chalcones is extended in two branches: their biological activity, including anti-inflammatory [1], antimitotic [2], anti-leishmanial [3], anti-invasive [4], anti fungal [5], antimalarial [6] and anti-tumor [7] properties; as well as their recognized synthetic utility in the preparation of pharmacologically interesting heterocyclic systems such as thiazines, pyrimidines, and pyrazoles. Here we are reporting a novel bis-chalcone prepared from 3-acetyl-2,5-dimethylfuran and terephthalaldehyde. The product is assumed to exist as one *E,E*-diastereomer, since in the ¹H-NMR spectrum the olefinic protons display coupling constants of 15.6 Hz indicative of the *E*-configuration.

Figure 1. Synthesis of compound 3.



A solution of 3-acetyl-2,5-dimethylfuran (2.34 mL, 0.028 mol) and terephthalaldehyde (2.0 g, 0.014 mol) in an ethanolic solution of NaOH (6.0 g in 10 mL of ethanol) was stirred for 20 h at room temperature. The solution was poured into ice cold water of pH~2 (pH adjusted by HCl). The solid was separated and dissolved in CH_2Cl_2 , washed with a saturated solution of NaHCO_3 and evaporated to dryness. The residue was recrystallized from methanol/chloroform to give a yellow solid: Yield: 78%; m.p. 182 °C.

EI-MS m/z (rel. int.%): 376 (76) $[\text{M}+1]^+$,

IR (KBr) $\nu_{\text{max}} \text{ cm}^{-1}$: 2956 (C-H), 1655 (C=O), 1567 (C=C).

^1H NMR (600 MHz, CDCl_3) δ : 7.72 (d, 2H, $J = 15.6$ Hz, C=CH), 7.22 (d, 2H, $J = 15.6$ Hz, CO=CH), 7.63 (s, 4H, Ar-H), 6.34 (s, 2H, 2 $\times$ furan-H), 2.62 (s, 6H, 2 $\times$ CH₃), 2.30 (s, 6H, 2 $\times$ CH₃).

^{13}C NMR (150 MHz, CDCl_3) δ : 185.65, 158.20, 150.17, 141.60, 136.70, 128.99, 125.00, 122.41, 105.54, 14.53, 13.05.

Anal. calc. for $\text{C}_{24}\text{H}_{22}\text{O}_4$: C, 76.99, H, 5.92, O, 17.09; Found: C, 76.95, H, 5.88, O, 19.98.

Acknowledgements

The authors would like to thank the deanship of scientific research for the financial support of this work via Grant No. (3-045/430).

References

- Vogel, S.; Barbic, M.; Jurgenliemk, G.; Heilmann, J. Synthesis, cytotoxicity, anti-oxidative and anti-inflammatory activity of chalcones and influence of A-ring modifications on the pharmacological effect. *Eur. J. Med. Chem.* **2010**, *45*, 2206-2213.
- Kamal, A.; Balakrishnan, G.; Ramakrishna, G.; Shaik, T.B.; Sreekanth, K.; Balakrishna, M.; Rajender, D.; Kalivendi, S.V. Synthesis and biological evaluation of cinnamido linked pyrrolo[2,1-c][1,4]benzodiazepines as antimitotic agents. *Eur. J. Med. Chem.* **2010**, *45*, 3870-3884.

3. Reichwald, C.; Shimony, O.; Sacerdoti-Sierra, N.; Jaffe, C.L.; Kunick, C. A new Heck reaction modification using ketone Mannich bases as enone precursors: Parallel synthesis of anti-leishmanial chalcones. *Bioorg. Med. Chem. Lett.* **2008**, *18*, 1985-1989.
4. Katritzky, A.R.; Kuanar, M.; Dobchev, D.A.; Vanhoecke, B.W.A.; Karelson, M.; Parmar, V.S.; Stevens, C.V.; Bracke, M.E. QSAR modeling of anti-invasive activity of organic compounds using structural descriptors. *Bioorg. Med. Chem.* **2006**, *14*, 6933-6939.
5. Hussain, T.; Siddiqui, H.L.; Zia-ur-Rehman, M.; Yasinzi, M.M.; Parvez, M. Anti-oxidant, anti-fungal and anti-leishmanial activities of novel 3-[4-(1*H*-imidazol-1-yl) phenyl]prop-2-en-1-ones. *Eur. J. Med. Chem.* **2009**, *44*, 4654-4660.
6. Wu, X.; Wilairat, P.; Go, M. Antimalarial activity of ferrocenyl chalcones. *Bioorg. Med. Chem. Lett.* **2002**, *12*, 2299-2302.
7. Rao, Y.K.; Fang, S.; Tzeng, Y. Synthesis, growth inhibition, and cell cycle evaluations of novel flavonoid derivatives. *Bioorg. Med. Chem.* **2005**, *13*, 6850-6855.

© 2010 by the authors; licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution license (<http://creativecommons.org/licenses/by/3.0/>).