

## Synthesis of 4-(4-(dimethylamino)phenyl)-5-acetyl-6-phenyl-3,4-dihydropyrimidin-2(1H)-thione

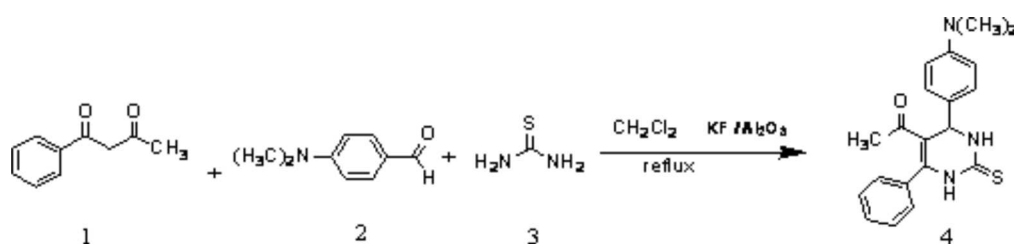
Fatma Aydin

Canakkale Onsekiz Mart University, Department of Chemistry,  
17020, Canakkale-Turkey

e-mail: [faydin@comu.edu.tr](mailto:faydin@comu.edu.tr)

Received: September 2005 / Accepted: 13 October 2005 / Published: 28 February 2006

**Keywords:** Biginelli reaction, KF/Al<sub>2</sub>O<sub>3</sub>, Benzoyacetone, thiourea



4-(4-(dimethylamino)phenyl)-5-acetyl-6-phenyl-3,4-dihydropyrimidin-2(1H)-thione was prepared from benzoyacetone (**1**) (1.62 g, 10 mmol), N,N-dimethylaniline (**2**) (1.21 g, 10 mmol) and thiourea (**3**) (1.140 g, 15 mmol) in dichloromethane (15 mL) as solvent using KF/Al<sub>2</sub>O<sub>3</sub> (5 g) as a catalysis. The reaction mixture was heated under reflux conditions and stirred by using magnetic stirring until the one-pot addition was completed. The crude was filtered to remove residue on KF/Al<sub>2</sub>O<sub>3</sub> and washed with DCM. The crude product was purified by column chromatography (silica gel, ethylacetate/*n*-hexane, 4:1, V/V as eluant) to give 4-(4-(dimethylamino)phenyl)-5-acetyl-6-phenyl-3,4-dihydropyrimidin-2(1H)-thione as pure yellow (**4**) (2.98 g, 85% yield).

Melting point: 118-120°C

UV (EtOH; λ<sub>max</sub> nm): 340

IR (KBr, cm<sup>-1</sup>): 3200 (N-H); 2951 (C-H Ar); 2853 (C-H); 2253; 1624; 1220

<sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>): δ= 3.48 (6H, s, N(CH<sub>3</sub>)<sub>2</sub>); 3.00 (1H, s, Benzyl-H); 2.48 (2H, brs, NH); 1.607 (3H, s, CH<sub>3</sub>CO); 6.8 (2H, d, Ar-H); 7.1-7.3 (5H, m, Ar-H); 7.8 (2H, d, Ar-H).

<sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>): δ= 190 (C=O); 184 (C=S); 175 (=C-N); 170 (-C-N); 154, 132, 129, 127, 125, 111, 40, 25

MS (m/z): 351 [M<sup>+</sup>]; 350; 308; 231.

Elemental Analysis: Calculated for C<sub>20</sub>H<sub>21</sub>N<sub>3</sub>OS: C, 68.41%; H, 6.08%; N, 11.89%; O, 4.58%; S, 9.17%. Found: C, 68.35%; H, 6.03%; N, 11.95%; O, 4.55%; S, 9.12%.

### References

1. Kappe, C. O., *Tetrahedron*, **1993**, *49*, 6937-6963
2. Kappe, C. O., *Molecules*, **1998**, *3*, 1-9

3. Lin, H.; Ding, J.; Chen, X. and Zhang, Z. *Molecules* **2000**, *5*, 1240-1243
4. Salehi, P.; Dabiri, M.; Zolfigol, M. A. and Fard, M. A. B. *Tetrahedron Letters* **2003**, *44*, 2889-2891.

*Sample Availability:* Available from MDPI.

© 2006 [MDPI](http://www.mdpi.org). All rights reserved.