

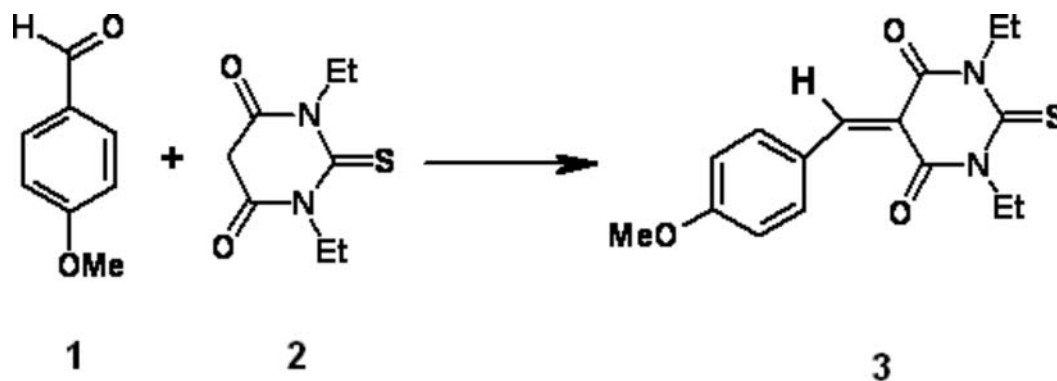
1,3-Diethyl-5-(4-methoxybenzylidene)-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione**Abdullah Mohamed Asiri^{a*}, Khaled Ahmed Alamry^a Abraham F. Jalbout^{b,c} and S Zhang^b**

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1,3-Diethyl-5-(4-methoxybenzylidene)-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione **3** was prepared by Knoevenagel condensation of 4-methoxybenzaldehyde **1** and N,N-diethylthiobarbituric acid **2** in ethanol using piperidine as a base [1,2].

N,N-Diethylthiobarbituric acid **2** (7.5 g, 0.0375 mol) and 4-methoxybenzaldehyde **1** (5.1 g, 0.0375 mmol) in ethanol (75 mL) were heated under reflux for five minutes. Piperidine (1.0 mL) was added in one portion and the reflux was continued for five hours. The reaction mixture was cooled to room temperature and the solid formed was filtered, washed with cold ethanol (2x 40 mL) and dried. 1,3-Diethyl-5-(4-methoxybenzylidene)-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione **3** was recrystallized from ethanol as yellow crystals (10.86g, 91%).

M.p. 147 °C (uncorrected.).

UV $\lambda_{\max}$ (nm; CHCl₃)/ ϵ (dm³.mol⁻¹.cm⁻¹) 414/25000 and 258/ 5848.

IR (KBr) cm^{-1} 2978, 1661 (C=O), 1602 (C=C), 1433.5, 1387 (CS).

Theoretical (cm^{-1} ; AM1): 1650.6; 1462.1; 1388.8

^1H -NMR (400 MHz; CDCl_3 ; Me_4Si) δ 8.52 (1H, s, -CH=C), 8.37 (2H, d, $J = 8.94$ Hz, aromatic), 7.00 (2H, d, $J = 7.24$ Hz, $J = 8.94$ Hz, aromatic), 4.58 (4H, t, $2\times\text{CH}_2$), 3.90 (3H, s, MeO), 1.32 (6H, q, CH_3).

^{13}C -NMR (100 MHz; CDCl_3 ; Me_4Si) δ 174.9 (C=S), 163.8, 163.4 (C=O), 157.7, 137.7 (CH oliefinc), 155.6, 132.4, 128.2, 126.5 (C), 114.5, 56.03 (MeO), 44.6, 44.3 (CH_2), 13.5, 13.2 (CH_3).

Anal.Calc. for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_3\text{S}$ (318.392): C 60.36, H 5.70, N 10.07; found : C 60.15, H 5.5.79, N 10.20.

Theoretical calculations were performed with the GAUSSIAN 98 package [3] using the AM1 semi-empirical model [4]. From the above (AM1 values) we see that the theoretical and experimental values are in good agreement. Using a rigid-rotor/harmonic-oscillator (RRHO) approximation in agreement with NIST standards we compute at 298.15 K, S (entropy in $\text{J mol}^{-1} \text{K}^{-1}$) as 674.66, C_p as 329.70 (heat capacity at constant pressure in $\text{kJ mol}^{-1} \text{K}^{-1}$) and $H^\circ - H^\circ_{298.15}$ as 58.50 (enthalpy content, in kJ mol^{-1}). The equations most useful for indexing these species with the NIST are:

	<i>Fitted Thermodynamic Equation ($T/1000=t$)</i>	T_1	T_2	T_3
S	$-7.63127*\ln(t) + 1284.11508*t - 578.09841*t^2/2 + 33.99402*t^3/3 + 0.57583/(2*t^2) + 311.06252$	425.38	674.66	1317.25
C_p	$329.41584 + 1243.04945*t - 253.18884*t^2 - 1.8789*t^3 - 0.25805*t^{-2}$	157.48	329.70	738.27
DH	$148.67084*t + 5284.44948*t^2/2 - 9023.3457*t^3/3 + 4547.86803*t^4/4 + 16.23129$	10.27	58.50	460.39

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