

Short Note

5-[(3,5-Dimethyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-1,3-diethyl-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione

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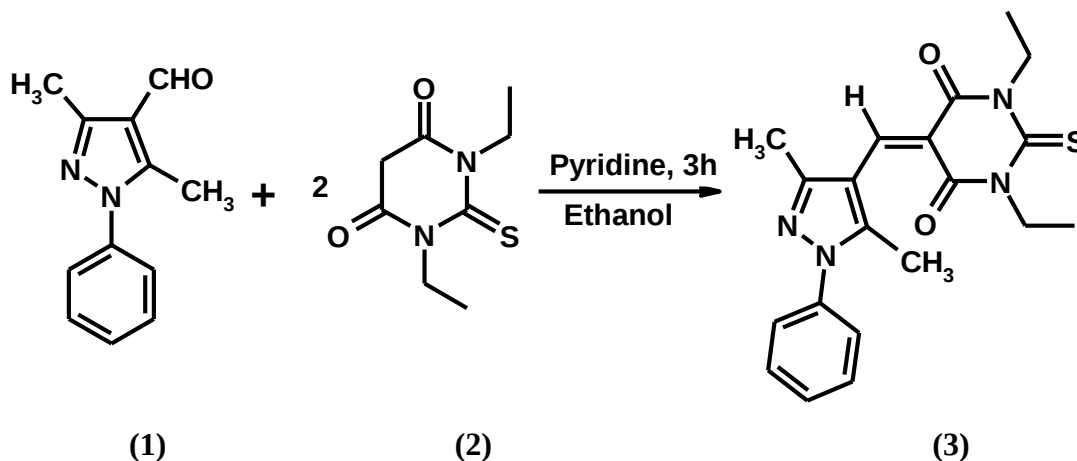
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Abstract: The title compound, 5-[(3,5-dimethyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-1,3-diethyl-2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione, has been synthesized by condensation of 1,3-diethyl-2-thiobarbituric acid and 3,5-dimethyl-1-phenylpyrazole-4-carbaldehyde in ethanol in the presence of pyridine. The structure of this new compound was confirmed by elemental analysis, IR, ¹H-NMR, ¹³C-NMR and EI-MS spectral analysis.

Keywords: thiobarbituric acid; Knoevenagel condensation; pyridine

The Knoevenagel condensation of aldehydes with active methylene compounds is an important and widely employed method for donor-acceptor chromophore formation in organic synthesis [1] with numerous applications in the synthesis of fine chemicals such as photoelectronics [2], photophotonics, photodynamic therapy [3], electrochemical sensing [4], optical limiting [5], Langmuir film and photoinitiated polymerization [6]. The donor acceptor chromophores are also applicable in the field of biomedical chemistry. Due to the wide application of donor-acceptor chromophores, the authors have undertaken the synthesis of a novel donor-acceptor chromophore.



A mixture of 1,3-diethyl-2-thiobarbituric acid (1) (1.0 g, 0.005 mol), 3,5-dimethyl-1-phenylpyrazole-4-carboxaldehyde (2) (1.0 g, 0.005 mol) and a few drops of pyridine in anhydrous ethanol (15 mL) was refluxed at 80 °C for 3 h with continuous stirring. Progress of the reaction was monitored by TLC. After completion of the reaction, the solution was cooled. The heavy precipitate thus obtained was collected by filtration and purified by recrystallization from methanol/chloroform to give the title compound (3).

Yield: 78%; m.p. 177–178 °C.

EI-MS m/z (rel. int.%): 383 (62) $[M+1]^+$.

IR (KBr) $\nu_{\max}$ cm^{-1} : 2979 (C-H_{aromatic}), 2926 (C-H_{aliphatic}) 1694 (C=O), 1568 (C=C), 1194 (C-N), 1172 (C=S).

$^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 8.56 (s, C=CH), 7.52 (d, $J = 3.6$ Hz, CH_{aromatic}), 7.50 (d, $J = 1.8$ Hz, CH_{aromatic}), 7.45 (dd, $J = 3.0, 2.8$ Hz, CH_{aromatic}), 7.43 (dd, $J = 1.2, 1.8$ Hz, CH_{aromatic}), 7.24 (dd, $J = 1.4, 1.6$ Hz, CH_{aromatic}) 4.56 (t, $J = 3.6$ Hz, CH₃-CH₂-N), 2.37 (s, -CH₃), 2.28 (s, -CH₃), 1.34 (q, $J = 6.0$ Hz, CH₃-CH₂-N),

$^{13}\text{C-NMR}$ (600 MHz, CDCl_3) δ : 178.94, 160.84, 158.29, 152.91, 150.40, 145.19, 138.46, 129.34, 128.69, 125.04, 117.63, 114.45, 43.92, 43.50, 14.63, 13.55, 12.70, 12.43.

Anal. calc. for $\text{C}_{20}\text{H}_{22}\text{O}_2\text{N}_4\text{S}$: C, 62.82, H, 5.75, N, 14.65. Found: C, 62.76, H, 5.55, N, 14.58.

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References and Notes

- Asiri, A.M. Synthesis and characterization of dyes exemplified by 2-arylidene-dicyanomethyleneindane. *Dyes Pigm.* **1999**, 42, 209–213.

2. Jiang, L.; Chang, Q.; Ouyang, Q.; Liu, H.; Wang, Y.; Zhang, X.; Song, Y.; Li, Y. Fabrication and nonlinear optical properties of an ultrathin film with acceptor–donor periodically overlapping structure. *Chem. Phys.* **2006**, *324*, 556–562.
3. Wu, C.; Tretiak, S.; Chernyak, V.Y. Excited states and optical response of a donor–acceptor substituted polyene: A TD-DFT study. *Chem. Phys. Lett.* **2007**, *433*, 305–311.
4. Chandrasekarn, Y.; Dutta, G.K.; Kanth, R.B.; Patil, S. Tetrahydroquinoxaline based squaraines: Synthesis and photophysical properties. *Dyes Pigm.* **2009**, *83*, 162–167.
5. Liu, Y.; Zhou, J.; Zhang, X.; Liu, Z.; Wan, X.; Tian, J.; Wang, T.; Chen, Y. Synthesis, characterization and optical limiting property of covalently oligothiophene-functionalized graphene material. *Carbon* **2009**, *47*, 3113–3121.
6. Bosch, P.; Peinado, C.; Martin, V.; Catalina, F.; Corrales, T. Fluorescence monitoring of photoinitiated polymerization reactions: Synthesis, photochemical study and behaviour as fluorescent probes of new derivatives of 4'-dimethylaminostyryldiazines. *J. Photochem. Photobio. A: Chem.* **2006**, *180*, 118–129.

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