

Short Note

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

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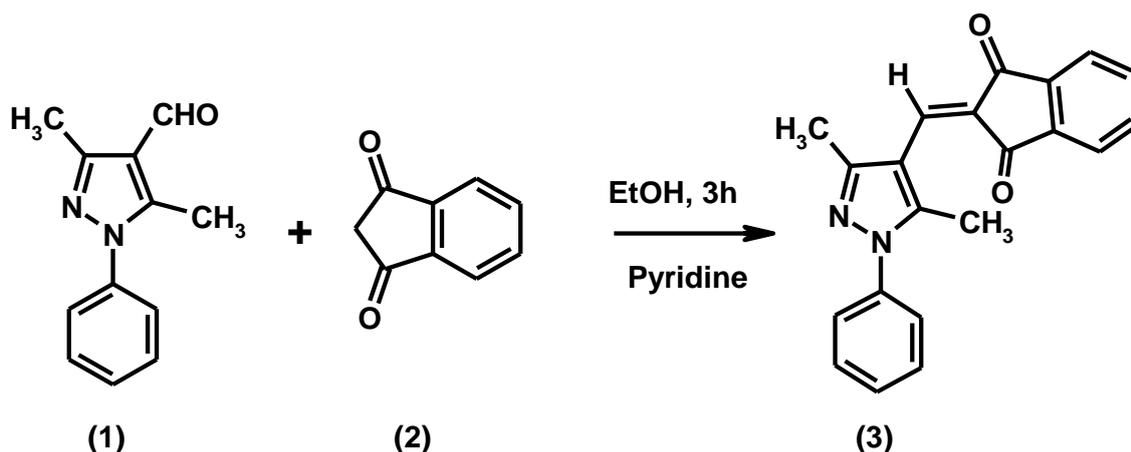
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Abstract: The title compound 2-[(3,5-dimethyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-indane-1,3-dione (**3**) was synthesized in high yield by reaction of 3,5-dimethyl-1-phenyl-pyrazole-4-carbaldehyde and indane-1,3-dione in ethanol in the presence of pyridine. The structure of this new compound was confirmed by elemental analysis, IR, ¹H NMR, ¹³C NMR and GC-MS spectral analysis.

Keywords: Knoevenagel condensation; indane-1,3-dione; pyridine

Naturally occurring as well as synthetic pyrazole-containing heterocyclic compounds have great importance for their biological activities such as anti-bacterial [1], anti-inflammatory [2], anti-hypertensive [3], anti-cancer [4], and anti-amoebic activity [5]. Pyrazole-containing donor-acceptor chromophores are also applicable in materials fields for their properties such as non-linear optical (NLO), optical limiting [6], electrochemical sensing [7] and langmuir film [8]. Due to the wide application of pyrazoles we decided to synthesize a new pyrazole-containing donor-acceptor chromophore by Knoevenagel condensation in analogy to a previously published procedure [9].

Scheme 1. Synthesis of compound 3.



Experimental

Materials

3,5-Dimethyl-1-phenylpyrazole-4-carbaldehyde and indane-1,3-dione were purchased from Aldrich Chemicals. Melting points of the synthesized compounds were determined in open-glass capillaries on a Stuart-SMP10 melting point apparatus and are uncorrected. IR absorption spectra were recorded in the 4,000–400 cm^{-1} range on a Shimadzu FTIR-8400s using KBr pellets, ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker-AVANCE-III 600 spectrometer at 600 MHz and 150 MHz, respectively, chemical shifts are reported as parts per million (ppm) downfield from TMS (Me_4Si) used as an internal standard.

A mixture of 3,5-dimethyl-1-phenylpyrazole-4-carbaldehyde (1) (1.0 g, 0.005 mol), indane-1,3-dione (2) (0.73 g, 0.005 mol) and a few drops of pyridine in ethanol (15 mL) was heated for 3 h. The progress of the reaction was monitored by TLC. The solid that separated from the cooled mixture was collected and recrystallized from a methanol-chloroform mixture to give the title compound (3) as a yellow solid.

Yield: 85%; m.p. 196–197 °C

GC-MS m/z (rel. int. %): 330 (72) $[\text{M}+1]^+$

IR (KBr) ν_{max} cm^{-1} : 3035 (Ar-H), 2859 (C-H), 1663 (C=O), 1578 (C=C)

^1H NMR ($\text{DMSO}-d_6$) (δ /ppm): 8.02 (d, $J = 2.8$ Hz), 7.97 (d, $J = 2.8$ Hz), 7.93 (s, C=CH), 7.82 (dd, $J = 2.8, 2.8$ Hz), 7.54 (m, 5H, Ar-H), 7.46 (dd, $J = 6.6$ Hz) 2.44 (s, CH_3), 2.36 (s, CH_3)

^{13}C NMR (CDCl_3) δ : 190.33, 188.65, 152.11, 144.83, 142.56, 140.10, 138.84, 136.16, 135.02, 134.76, 129.26, 128.11, 126.71, 125.07, 123.11, 122.86, 123.11, 122.86, 116.14, 14.14, 13.04.

Anal. calc. for $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2$: C, 76.81, H, 4.91, N, 8.53. Found: C, 76.79, H, 4.88, N, 8.84.

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