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Short Note

N-[1-(1*H*-Benzimidazol-2-yl)-2-(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)vinyl]benzamide

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Abstract: A novel benzimidazole derivative (**2)** was synthesized by hydrolyzing 4-[(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-2-phenyloxazol-5(4*H*)-one **(1)**, the azlactone precursor in an acidic medium and treating the product with *o*-phenylenediamine (OPDA) *in situ.* The structure of the title compound (**2)** was established on basis of FT-IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Keywords: benzimidazole; oxazolone; *o*-phenylenediamine

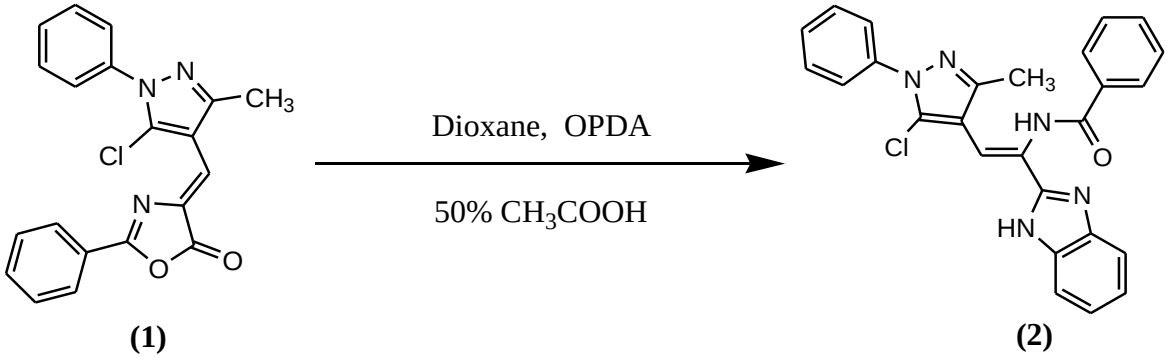
1. Introduction

The benzimidazole nucleus is an important heterocyclic ring, because of its synthetic utility and broad range of pharmacological activities. The benzimidazole ring, containing two nitrogen atoms, plays an important role in the constitution of many of the natural products as well as synthetic compounds. Benzimidazole derivatives possess various pharmacological activities including antibacterial [1], anti-fungal [2], antiviral [3], antiparasitic [4], antiprotozoal [5], antihelmintic [6], anti-inflammatory [7] anti-ulcer [8], anti-hypertensive [9], anticonvulsant [10] and CNS activity [11].

2. Results and Discussion

The title compound (**2**) was synthesized as presented in Scheme 1 by hydrolyzing compound **(1)**, the azlactone precursor in 50% acetic acid medium and treating the product with *o*-phenylenediamine (OPDA) *in situ*. Erlenmeyer-Plochl azlactone synthesis was utilized for the synthesis of the azlactone from hippuric acid [12] and 5-chloro-3-methyl-1-phenyl-1*H*-pyrazole-4-carboxaldehyde. The util;ized carboxaldehyde was in turn synthesized *via* Vilsmeier-Haack reaction.

Scheme 1. Synthetic route to the title compound.



3. Experimental

The melting point was determined in an open-end capillary tube on a digital melting point apparatus and is uncorrected. Infrared (IR) and proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a Nicolet 380 FT-IR (KBr) and a Bruker DRX-300 instrument, respectively. Chemical shifts are expressed in parts per million (ppm) relative to tetramethylsilane as an internal standard. The elemental analysis was performed on a Vario EL III CHNS analyzer using sulphanilic acid as a standard. The ESI-MS spectrum was recorded on a Waters Micromass Q-TOF Micro. The homogeneity of the compounds was monitored by ascending thin-layer chromatography (TLC), visualized by iodine vapour.

*Synthesis of N-[1-(1H-benzimidazol-2-yl)-2-(5-chloro-3-methyl-1-phenyl-1H-pyrazol-4-yl)vinyl]benzamide (**2**)*

To an equimolar quantity *i.e*. (0.004 mol) of the compound **(1)** and *o*-phenylendiamine (OPDA), was added 20 mL of 50% acetic acid followed by 30 mL of dioxane until the entire solid was dissolved. Then, the solution was refluxed at 135–140 ℃ for 20 h with stirring. The reaction mixture was then allowed to cool and it was evaporated nearly to dryness. This resulted in brownish material which was then redissolved in methanol, followed by addition of activated charcoal and then it was further refluxed for another 10 min, then filtered to remove any traces of activated charcoal and further allowed to cooled and the solid thus separated was filtered, washed and then recrystallised from ethanol to give pure crystals.

Yield: 72%; mp: 244–248 ℃; R_f: 0.2, [toluene/ethyl acetate/formic acid (5:4:1)]; buff white crystalline solid.

IR (KBr) ν_{max} (cm^{-1}): 3153 (N-H stretching), 3056 (aromatic C-H stretching), 2926 (aliphatic C-H stretching), 2851, 1654 (CONH), 1611 (C=N), 1577 (C=C).

^{1}H-NMR (300 MHz, DMSO-d_6): δ (ppm) 12.55 (s, 1H, CON*H*, *D*$_2$*O* exchangeable), 10.17 (s, 1H, N*H*, *D*$_2$*O* exchangeable), 8.05–8.03 (d, *J* = 7.2 Hz, 2H, Ar-*H*), 7.60–7.44 (m, 10H, Ar-*H*), 7.20–7.17 (d, *J* = 9.3 Hz, 2H, Ar-*H*), 7.11 (s, 1H, C*H*=C), 2.17 (s, 3H, C*H*$_3$).

^{13}C-NMR (75 MHz, DMSO-d_6): δ (ppm) 163.6 (*C*=O), 151.10 (*C*-CH_3), 148.66 (C_2), 143.92 (CH=*C*-N), 138.04, 135.15, 134.36, 132.24, 129.77, 128.75, 128.46, 128.07, 129.00, 122.88, 122.07, 119.13, 114.94, 111.80, 14.29 (CH_3).

Anal. Calcd. for $C_{26}H_{20}ClN_5O$: C, 68.80; H, 4.44; N, 15.43; Found: C, 68.97; H, 4.68; N, 15.61.

ESI-MS: *m/z* = 454.2 (M^+, 100%), 456.1 (M^++2, 36%).

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