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Short Note

1-(8-Chloro-3-methyl-1*H*-pyrazolo[4,3-*c*]cinnolin-1-yl)-2-(2-chlorophenyl)ethanone

Sandhya Bawa, Rajiv K. Tonk *, Gita Chawla, Suresh Kumar and Obaid Afzal

Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Jamia Hamdard (Hamdard University), New Delhi-110062, India

* Author to whom correspondence should be addressed; E-Mail: rajiv_tonk@rediffmail.com.

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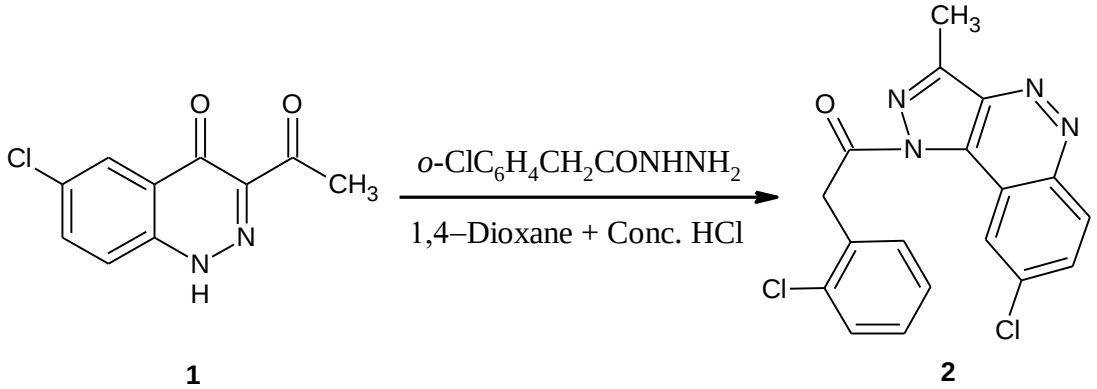
Abstract: 1-(8-Chloro-3-methyl-1*H*-pyrazolo[4,3-*c*]cinnolin-1-yl)-2-(2-chlorophenyl)ethanone **2** has been synthesized through condensation of 3-acetyl-6-chloro-1*H*-cinnolin-4-one **1** with 2-(2-chlorophenyl)acetohydrazide in polar aprotic solvent containing catalytic amount of conc. HCl. The structure of the title compound **2** was established on the basis of IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Keywords: cinnoline; pyrazole; 2-(2-Chlorophenyl)acetohydrazide

Cinnoline (or 1,2-diazanaphthalene) has obvious potential as antibacterial agent and along with other heterocyclic moieties showed a wide spectrum of pharmacological activities like antimicrobial, anti-inflammatory, analgesic, anticancer, antihypertensive and neuroleptic properties [1-5]. In continuation of our previous work on diazines and benzfused heterocyclic compounds [6-8], we are reporting herein the synthesis of 1-(8-Chloro-3-methyl-1*H*-pyrazolo[4,3-*c*]cinnolin-1-yl)-2-(2-chlorophenyl)ethanone **2**.

In the ^{1}H-NMR spectrum ($CDCl_3$ solvent) of compound **2**, the characteristic singlet of cinnoline-NH proton (D_2O exchangeable) in compound **1** at 13.90 ppm disappeared, and the singlet of methyl group (-$COCH_3$) shifted upfield to 2.36 ppm along with a new signal of methylene group (–CH_2-) at 4.39 ppm. Moreover, the cyclic carbonyl carbon signal disappeared in the ^{13}C-NMR spectrum of compound **2**. The fact was also supported by the mass spectrum of compound **2** which showed molecular ion peak (M^+) at m/z 370.04 along with two characteristic peaks at 372.04 (M^++2) and 374.04 (M^++4). The values are in complete agreement with the structure assigned.

Scheme 1. Synthetic route for the title compound **2**.



1 **2**

Synthesis of 1-(8-Chloro-3-methyl-1H-pyrazolo[4,3-c]cinnolin-1-yl)-2-(2-chlorophenyl)ethanone **2**

Accurately weighed quantities of **1** (0.5 g, 2 mmol) and 2-(2-Chlorophenyl)acetohydrazide (2.5 mmol) were mixed on a magnetic stirrer for 0.5 h in anhydrous 1,4-dioxane (25 mL) containing 0.2 mL of concentrated hydrochloric acid. The resulting mixture was boiled under reflux for 14 h. After completion of reaction (monitored by TLC) the reaction mixture was allowed to cool, concentrated under reduced pressure and poured in to ice-cold water with constant stirring. The product obtained was filtered, dried and then purified through column chromatography (Hexane/AcOEt 4:1) to afford the titled compound **2**.

Yield 56%, m.p. 206–208 ℃, Yellow amorphous solid.

IR (KBr) cm^{-1}: 1664 (C=O), 1593 (C=N), 1569 (C=C), 1263, 1166 (-C-N=N-), 746 (C-Cl).

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 2.36 (s, 3H, CH_3), δ 4.39 (s, 2H, CH_2), 7.23–7.32 (m, 4H, H_{Phenyl}), δ 7.39 (br. s, 3H, $H_{Cinnoline}$).

^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 2.25 (s, 3H, CH_3), δ 4.35 (s, 2H, CH_2), δ 7.27–7.32 (m, 2H, $H_{Cinnoline}$), 7.39–7.52 (m, 4H, H_{Phenyl}), δ 7.66 (br s, 1H, $H_{Cinnoline}$).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 12.0 (CH_3), 67.0 (CH_2), 116.7, 117.2, 126.8, 128.7, 129.4, 129.8, 131.5, 131.8, 134.7, 139.2, 151.2, 159.0, 166.6 (C=O).

DART-MS m/z: 370.04 (M^+), 372.04 (M^++2), 374.04 (M^++4).

Anal. Calcd for $C_{18}H_{12}Cl_2N_4O$: C, 58.24; H, 3.26; N, 15.09. Found: C, 58.44; H, 3.31; N, 15.04.

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