

Short Note

(8-Chloro-3-methyl-1*H*-pyrazolo[4,3-*c*]cinnolin-1-yl) (pyridin-4-yl)methanone

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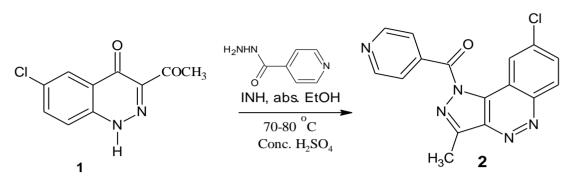
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Abstract: (8-Chloro-3-methyl-1*H*-pyrazolo[4,3-*c*]cinnolin-1-yl) (pyridin-4-yl)methanone **2** has been synthesized through condensation of 3-acetyl-6-chloro-1*H*-cinnolin-4-one **1** with isonicotinic acid hydrazide (INH) in absolute ethanol. The structure of the title compound **2** was established on the basis of IR, ¹H-NMR, ¹³C-NMR and mass spectral data.

Keywords: cinnoline; pyrazole; isonicotinic acid hydrazide

Cinnoline is a benzfused pyridazine containing two nitrogen atoms at 1,2-position and it is categorized under benzfused diazines class of heterocyclic compounds. Literature shows several compounds containing a cinnoline moiety with a wide spectrum of pharmacological activities, like antimicrobial, anti-inflammatory, anticancer, antimalarial and antipsychotic *etc.* [1–5]. As a part of research on benzfused heterocyclic compounds [6], we report here the synthesis of (8-chloro-3-methyl-1H-pyrazolo[4,3-*c*]cinnolin-1-yl) (pyridin-4-yl)methanone **2**.

The ¹H-NMR spectrum of the compound **1** showed two singlets: one for the methyl group at 2.60 ppm and another for the cinnoline-NH proton at 13.90 ppm which is D₂O exchangeable. The characteristic peak of the cinnoline-NH proton disappeared and the singlet of the methyl group shifts to 2.34 ppm in the ¹H-NMR spectrum of compound **2**. Furthermore, in the ¹³C-NMR spectrum of compound **2** the peaks due to carbonyl carbons were missing. The fact was also supported by the mass spectrum of compound **2** which showed an (M + 2) peak at m/z 326. The values are completely in agreement with the structure assigned. Starting material 3-acetyl-6-chloro-1*H*-cinnolin-4-one (**1**) was synthesized based on a literature method [7].



Scheme 1. Synthetic route for the title compound 2.

Synthesis of (8-chloro-3-methyl-1H-pyrazolo[4,3-c]cinnolin-1-yl) (pyridin-4-yl)methanone 2

To a solution of **1** (1.0 g, 0.005 mol) in absolute EtOH (40 mL) isonicotinic acid hydrazide (0.68 g, 0.005 mol) and conc. H_2SO_4 (0.4 mL) were added. The reaction mixture was refluxed for 6 h, then the excess ethanol was distilled off and the mixture was allowed to cool to room temperature. The resulting product was poured into 150 mL of cold H_2O . The precipitate was filtered, air dried and recrystallised from methanol. The purity of compound **2** was checked by TLC, using benzene/acetone (8:2) as mobile phase and iodine (I₂) as visualizing agent.

Yield 84%; mp 162 ℃; pale yellow amorphous solid.

IR (KBr) cm⁻¹: 2921 (C-H), 1690 (C=O), 1623 (C=N), 1524 (C=C), 756 (C-Cl).

¹H-NMR (300 MHz, CDCl₃): δ (ppm) 2.34 (s, 3H, -CH₃), 7.39 (unresolved broad singlet, 3H, cinnoline-H), 7.51 (d, 2H, pyridine-H, J = 8.8 Hz), 7.80 (d, 2H, pyridine-H, J = 8.4 Hz).

¹³C-NMR (75 MHz, CDCl₃): δ 12.9 (CH₃), 123.4, 128.8, 130.9, 132.4, 133.2, 138.4, 140.8, 144.3, 149.2, 151.7, 189.8 (C=O).

FAB-MS m/z: 324.09 (M⁺), 326.09 (M + 2).

Anal. Calcd for C₁₆H₁₀ClN₅O: C, 59.36, H, 3.11, N, 21.63; Found: C, 59.53, H, 3.09, N, 21.71.

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