

Short Note

5-[(5-Chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-2-(4-nitrobenzoyl)-3-phenyl-2,5-dihydro-1,2,4-triazin-6(1*H*)-one

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Abstract: A novel 1,2,4-triazin-6-one derivative (**2**) was synthesized by the reaction of the oxazolone derivative **1** with 4-nitrobenzoic acid hydrazide in the presence of sodium acetate and glacial acetic acid. The title compound **2** was characterized on basis of IR, ¹H-NMR, ¹³C-NMR and mass spectral data.

Keywords: 1,2,4-triazin-6-one; 2-phenyloxazol-5(4*H*)-one; 4-nitrobenzoic acid hydrazide

1. Introduction

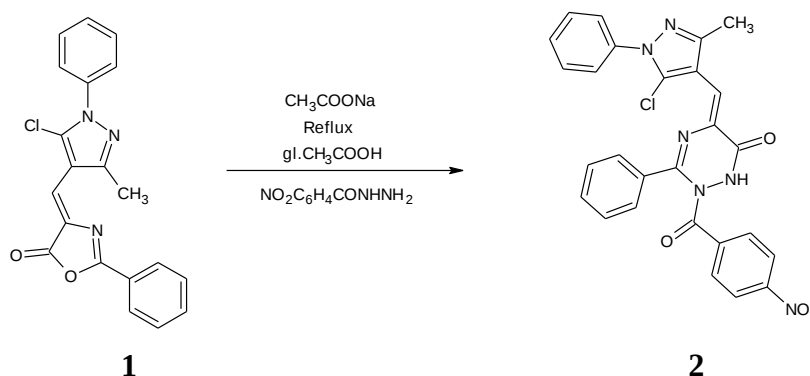
1,2,4-Triazine represents a class of heterocyclic compounds possessing significant biological activities which makes them targets for research in the field of medicine and agriculture [1-3]. 1,2,4-Triazines have gained considerable pharmacological interest due to their anticonvulsant [4,5], anticancer [6-9], antiprotozoal [10], anti-viral [11], anti-malarial [12], antibacterial [13-15] and antifungal effects [16-18]. In the field of agriculture, they showed effects such as insecticides, herbicides, plant growth regulators and they are deployed for enhancing crop yield [19-21].

2. Results and Discussion

The title compound **2** was synthesized by refluxing 4-[(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]-2-phenyloxazol-5(4*H*)-one **1** (see supplementary material) with 4-nitrobenzoic acid hydrazide in glacial acetic acid in the presence of sodium acetate, as presented in Scheme 1. The utilized azlactone was synthesized by the Erlenmeyer-Plochl method as discussed earlier [22]. The acid hydrazide was prepared from the acid *via* esterification followed by hydrazinolysis with hydrazine

hydrate. In the present reaction, the acid hydrazide acts as a nucleophile which attacks the carbonyl group of the oxazolone ring, followed by ring cleavage with concomitant cyclization to form the triazinone derivative **2** [5].

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 (^1H -NMR) spectra were recorded on a Nicolet 380 FT-IR (KBr) and a Bruker DRX-300 instrument, respectively. Chemical shifts are expressed in ppm relative to TMS as an internal standard. The elemental analysis was performed on a Vario EL III CHN 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 5-[(5-chloro-3-methyl-1-phenyl-1H-pyrazol-4-yl)methylene]-2-(4-nitrobenzoyl)-3-phenyl-1,2,4-triazin-6(5H)-one (2)

An equimolar quantity (*i.e.*, 0.01 mol) of compound **1** and of 4-nitrobenzoic acid hydrazide was refluxed along with sodium acetate (0.2 g) in glacial acetic acid (10 mL) for 6 h. The reaction mixture was then poured into crushed ice and stirred vigorously. The solid so obtained was filtered, washed with water, dried and recrystallised from ethanol.

Yield: 77%; m.p.: 242–244 °C; R_f : 0.75; mobile phase: toluene: ethyl acetate: formic acid (5:4:1); yellow crystalline solid.

IR (KBr) cm^{-1} : 3273 (N-H), 3052 (aromatic C-H), 2933 (aliphatic C-H), 1719 (CONH), 1665 (C=O), 1609 (C=N, imine), 1576 (C=C), 697 (C-Cl).

^1H -NMR (300 MHz, CDCl_3): δ (ppm) 10.58 (s, 1H, CONH, D_2O exchangeable), 8.17–8.15 (d, 2H, J = 8.7 Hz, Ar-H), 7.99–7.92 (m, 4H, Ar-H), 7.60–7.44 (m, 8H, Ar-H), 7.37 (s, 1H, CH=C), 2.82 (s, 3H, CH_3).

^{13}C -NMR (75 MHz, CDCl_3): δ 170.3 (C=O, benzoyl), 164.3 (C_6), 157.1 (C_3), 151.5 (C- NO_2), 150.2 (C- CH_3) 133.8, 132.1, 131.6, 129.2, 129.0, 128.9, 128.1, 127.5, 125.0, 123.8, 122.7, 114.1, 16.6 (C- CH_3).

Anal. Calcd. for $\text{C}_{27}\text{H}_{19}\text{ClN}_6\text{O}_4$: C, 61.54; H, 3.63; N, 15.94%; Found: C, 61.23; H, 3.89; N, 15.53%.

ESI-MS: m/z = 526.7 (M^+), 528.7(M^++2).

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