2-(2-Naphthoyl)-5-(furan-3-ylmethylene)-3-phenyl-1,2-dihydro-1,2,4-triazin-6(5*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 1-naphthoic acid hydrazide in the presence of sodium acetate and glacial acetic acid. The title compound 2 was characterized on basis of IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Keywords: 1,2,4-triazin-6-one; 4-(furan-2-ylmethylene)-2-phenyloxazol-5(4*H*)-one; 1-naphthoic acid hydrazide; Erlenmeyer-Plochl method.

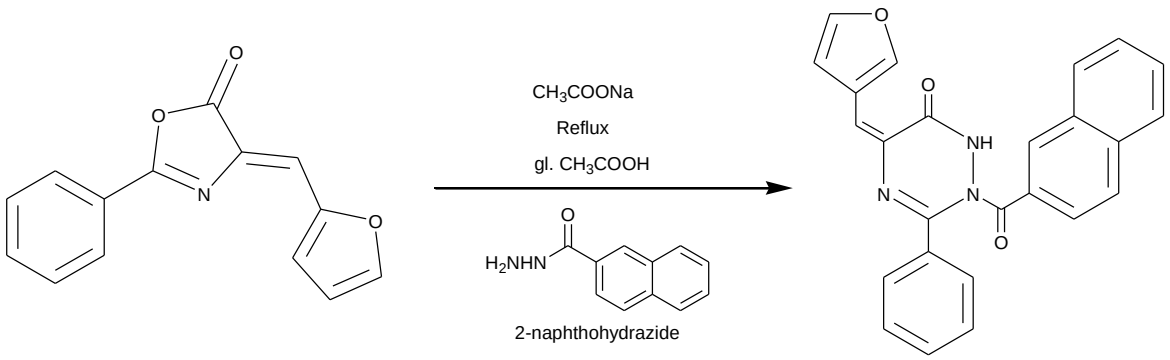
1. Introduction

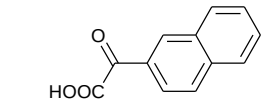
1,2,4-Triazine represents a class of heterocyclic compounds possessing significant biological activities which makes them targets for research in the field of medicine and agriculture [1-3]. 1,2,4-Triazines have gained considerable pharmacological interest due to their anticonvulsant [4-8], anticancer [9-14], antiprotozoal [15], anti-viral [16], anti-malarial [17, 18], antibacterial [19-22] and antifungal effects [23-26]. In the field of agriculture, they showed effects such as insecticides, herbicides, plant growth regulators and they are deployed for enhancing crop yield [27-29].

2. Results and Discussion

The title compound 2 was synthesized by refluxing 4-(furan-2-ylmethylene)-2-phenyloxazol-5(4*H*)-one 1 (see supplementary material) with 1-naphthoic 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 [30, 31]. 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.





2-(naphthalen-2-yl)-2-oxoacetic acid

1 2

3. Experimental

The melting point was determined in an open-end capillary tube on a digital melting point apparatus and is uncorrected. IR spectrum was acquired on an Agilent Infra Red Spectrometer, (model FTIR-cary 630). Both proton nuclear magnetic resonance (^{1}H-NMR) and ^{13}C NMR (DMSO) spectra of the synthesized compounds were performed with Bruker DRX-300 NMR Spectrometer at I.I.T, Delhi using $CDCl_3$ as solvent. Chemical shifts are expressed in ppm relative to TMS as an internal standard. Mass spectrum was recorded on a MicrOTOF-Q II at I.I.T, Delhi. The homogeneity of the compounds was monitored by ascending thin-layer chromatography (TLC), visualized by iodine vapour.

Synthesis of 2-(2-naphthoyl)-5-(furan-3-ylmethylene)-3-phenyl-1,2-dihydro-1,2,4-triazin-6(5H)-one (2)

An equimolar quantity (*i.e.*, 0.01 mol) of compound 1 and of 1-naphthoic 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: 80%; m.p.: 172–175 °C; Rf: 0.76; mobile phase: toluene: ethyl acetate: formic acid (5:4:1); brownish black crystalline solid.

IR cm^{-1}: 3176 (N-H), 3009 (aromatic C-H), 2933 (aliphatic C-H), 1724 (CONH), 1640 (C=O), 1580 (C=N, imine), 1525 (C=C).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) 10.29 (s, 1H, CON*H*, D_2O exchangeable), 8.01–7.98 (d, 1H, Ar-*H*), 7.78–7.69 (m, 3H, Ar-*H*), 7.55 (s, 1H, C*H*=C), 7.44-7.3 (m, 4H, Ar-*H*), 7.32-7.28 (m, 3H, Ar-*H*), 7.16-7.05 (m, 4H, Ar-*H*), 6.52-6.51 (s, 1H, Ar-*H*), 4.02-3.99 (s 1H, C*H*=C).

^{13}C-NMR (75 MHz, $CDCl_3$): δ 153.59 (*C*=O, benzoyl), 148.17 (C_6), 135.34 (C_3), 134.85 (2'-Furan), 134.60 (C_5-furan) 134.03, 131.91, 130.09, 129.33, 128.66, 128.41, 128.21, 128.03, 127.81, 126.86, 126.53, 124.99, 108.48 (C-CH_3).

ESI-MS: m/z = 407.1264(M^+), 408.1342(M^++2).

4. Conclusion

The new 1, 2, 4- triazin-6-one derivative, namely *2-(2-naphthoyl)-5-(furan-3-ylmethylene)-3-phenyl-1,2-dihydro-1,2,4-triazin-6(5H)-one* was successfully synthesized and characterized.

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