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Short Note

2-(Benzoylamino)-3-(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)acrylic acid

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Abstract: A novel acrylic acid derivative was synthesized *via* acid hydrolysis of 4-((5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene)-2-phenyloxazol-5(4*H*)-one in gl. acetic acid. The structure of the title compound was established on the basis of IR, ^{1}H NMR, ^{13}C-NMR and mass spectral data.

Keywords: acrylic acid; prop-2-enoic acid; substituted phenyl oxazolone

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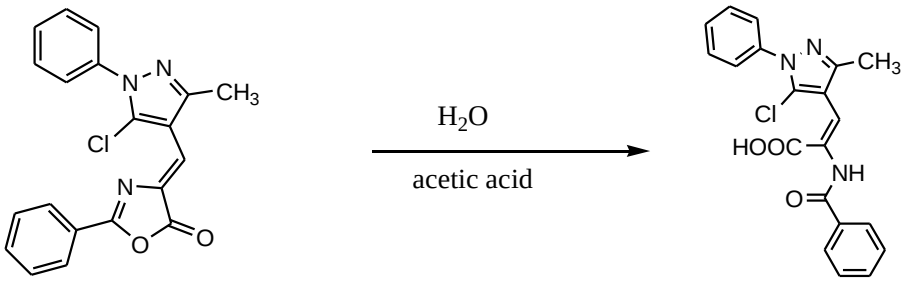
Introduction

Acrylic acids are highly reactive and essential building blocks in the production of the most commonly used industrial and consumer products [1,2]. Moreover, they have low temperature flexibility, excellent weathering and sunlight resistance, that's why they can be used in latex paint, coatings, textiles, adhesives, plastics, and a wide range of other properties [3,4]. Furthermore, polyacrylic acids are successfully used in the manufacture of hygienic products, detergents and waste water treatment chemicals [2].

The title compound **2** was synthesized *via* acid hydrolysis of 4-((5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl) methylene)-2-phenyloxazol-5(4*H*)-one **1** in acetic acid (Scheme 1).

Scheme 1. Synthetic route to the title compound.



1. 4-((5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene)-2-phenyloxazol-5(4*H*)-one

2. 3-(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-2-[(phenylcarbonyl) amino] prop-2-enoic acid

4-((5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl) methylene)-2-phenyloxazol-5(4*H*)-one **1** in turn was synthesized *via* Erlenmeyer-Plochl azalactone synthesis as reported in literature [5].

Experimental

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

*Synthesis of 2-benzamido-3-(5-chloro-3-methyl-1-phenyl-1H-pyrazol-4-yl) acrylic acid (**2**)*

0.001 moles of 4-((5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl) methylene)-2-phenyloxazol-5(4*H*)-one, water (5 mL) and acetic acid (20 mL) were refluxed for 15 h. The reaction mixture was kept in the refrigerator overnight. The solid obtained was filtered, washed with water, dried and recrystallised with the mixture of chloroform and methanol.

Yield: 87.5%; m.p.: 250–254 ℃; R_f: 0.5, mobile phase-Toluene: Ethyl acetate: Formic acid (5:4:1); off-white amorphous solid.

I.R. (KBr) cm^{-1}: 3243 (N-H stretching), 3023 (Aromatic C-H stretching), 2937 (Aliphatic C-H stretching), 1699 (C=O), 1646 (C=O), 1604 (C=N), 1523 (C=C), 690 (C-Cl).

^{1}H-NMR (300 MHz, DMSO-d_6): δ (ppm) 9.33 (s, 1H, COO*H*, D_2O exchangeable), 7.96–7.94 (d, 2H, Ar-*H*), 7.73 (s, 1H, N*H*CO, D_2O exchangeable), 7.52–7.40 (m, 8H, Ar-*H*), 7.15 (s, 1H, C*H*=C), 2.22 (s, 3H, -CH_3).

^{13}C-NMR (75 MHz, CDCl3): δ 167.81 (*C*ONH), 166.18 (*C*OOH), 148.98 (C_3-CH_3), 137.65, 133.56, 132.58, 129.85, 129.76, 129.01, 128.03, 126.96, 125.13, 120.63, 113.64, 14.01 (C_3-CH_3).

Anal. Calcd for $C_{20}H_{16}ClN_3O_3$: C, 62.91; H, 4.22; N, 11.01 Found: C, 62.58; H, 4.09; N, 10.93.

ESI-MS: *m/z* = 381.09 (M^+), 383.15 (M^++2).

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