Molbank **2010**, M652

OPEN ACCESS

molbank

ISSN 1422-8599

www.mdpi.com/journal/molbank

Short Note

6-Oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide

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Received: 17 November 2009 / Accepted: 22 December 2009 / Published: 26 January 2010

Abstract: We report herein the synthesis of 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide from β-benzoylpropionic acid and carbohydrazide by refluxing in absolute ethanol in presence of sodium acetate. The structure of the newly synthesized compound was established on the basis of IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data.

Keywords: pyridazinone; β-benzoylpropionic acid; carbohydrazide

1. Introduction

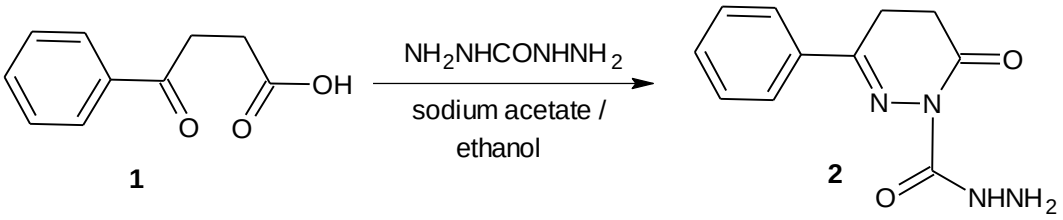
Pyridazines and their 3-oxo derivatives (*i.e.*, pyridazinones) are important 1,2-diazine-containing heterocyclic compounds having diverse pharmacological activity such as antihypertensive, antidiabetic, anti-inflammatory, antinociceptive and antimicrobial [1–6]. One of the general methods for the synthesis of these compounds involves condensation of 3-aroylpropionic or β-acetylacrylic acid with alkyl or arylhydrazines. Keeping in mind the medicinal utility of pyridazinones, we report herein the synthesis of 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide.

2. Results and Discussions

In the present study, synthesis of 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide **2** is reported from β-benzoylpropionic acid using carbohydrazide as a hydrazine derivative by refluxing in absolute ethanol in presence of anhydrous sodium acetate. The structure of compound **2** was established on the basis of IR, ^{1}H-NMR, ^{13}C-NMR and mass spectral data. In the ^{1}H-NMR spectrum of compound **2**, the peaks due to the –CONHNH$_2$ group were observed as a singlet at 9.78 ppm (CONH)

and another signal of NH_2 which overlaps with the multiplet of phenyl protons at 7.27–7.92 ppm. The two CH_2 groups of the pyridazinone ring appear as triplet-like signals at 2.35 and 2.87 ppm. Moreover, in the ^{13}C-NMR spectrum the signals due to the CH_2CH_2 group of the pyridazinone ring appear at 29.4 and 36.1 ppm, while the peaks due to the carbonyl carbon atoms (C=O) are observed at 169.8 and 178.3 ppm. The MS-(ESI) spectrum of compound **2** exhibits an M+1 peak at m/z = 233. The values are in complete agreement with the structure assigned.

Figure 1. Synthetic route to the title compound **2**.



3. Experimental

The starting material β-benzoylpropionic acid 1 was synthesized based on a literature method [7].

3.1. 6-Oxo-3-phenyl-5,6-dihydropyridazine-1(4H)-carbohydrazide **2**

To a solution of β-benzoylpropionic acid (0.01 mol) in absolute ethanol (30 mL) were added carbohydrazide (0.01 mol) and sodium acetate, and the mixture was refluxed for 6 h. After completion of the reaction, ethanol was distilled off and the residue was poured into cold water. The solid which separated was filtered and washed with water. The product was dried in air and crystallized from ethanol.

Yield, 85%; mp. 166–168 ℃; white amorphous solid.

IR (KBr) cm^{-1}: 3409, 3329, 3206, (N-H, str, symm. & asymm.), 3062 (CH), 1714 (C=O, amide I), 1629 (N-H, amide II), 1536 (C=C), 1078, 1052, 983, 837.

^{1}H-NMR (300 MHz, $CDCl_3$): δ 2.35 (t, 2H, CH_2), 2.87 (t, 2H, CH_2), 7.27–7.92 (m, 7H, Ar-H, NH_2, partially D_2O-exchangeable), 9.78 (s, 1H, CONH, D_2O-exchangeable).

^{13}C-NMR (75 MHz, DMSO-d_6) δ; 29.4 (CH_2), 36.1 (CH_2), 127.67 (CH), 128.3 (CH), 129.1 (CH), 130.6 (C), 149.2 (C=N), 169.8 (N-CO-N), 178.3 (C-CO-N).

Anal. Calcd for $C_{11}H_{12}N_4O_2$: C, 56.89; H, 5.21; N, 24.12. Found: C, 56.68; H, 5.26; N, 24.31. MS-(ESI); m/z 233 (M+1).

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Sample Availability: Sample of the compound **2** is available from authors.