

Short Note

6-Phenyl-2-(4-phenyl-5-thioxo-4,5-dihydro-1*H*-1,2,4-triazol-3-yl)-4,5-dihydropyridazin-3(2*H*)-one

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Abstract: 6-Phenyl-2-(4-phenyl-5-thioxo-4,5-dihydro-1*H*-1,2,4-triazol-3-yl)-4,5-dihydropyridazin-3(2*H*)-one **3** has been synthesized by a sequence of reactions starting from 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide **1**. The structure of the title compound **3** was established on the basis of IR, ¹H-NMR, ¹³C-NMR and mass spectral data.

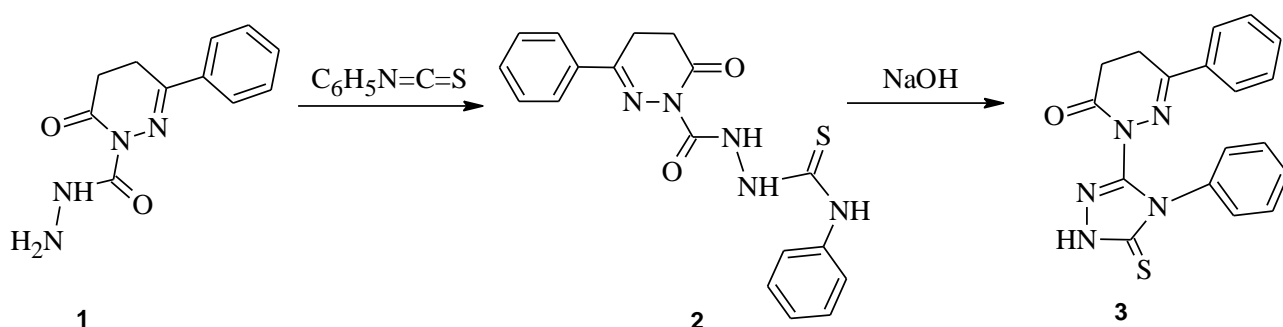
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The chemistry of 4,5-dihydro-3(2*H*)-pyridazinone has been an interesting field of study since decades. The synthesis of novel pyridazinone derivatives and investigation of their chemical and biological behavior have gained more importance in recent decades for biological, medicinal, and agricultural reasons. Living organisms have difficulties in the construction of N-N bonds that limits the natural abundance of compounds having such bonds. Pyridazinone derivatives, a class of compounds containing the N-N bond, exhibit a wide range of pharmacological activities such as antidepressant [1], antihypertensive [2,3], antithrombotic [4], anticonvulsant [5], cardiogenic [6], antibacterial [7], diuretic [8], anti-HIV [9] and anticancer [10]. Some pyridazinone derivatives like indolidan [11], bemoradan [12], primobendan [13], levosimendan [14] are already approved in the clinical market. The current work describes the synthesis of 6-phenyl-2-(4-phenyl-5-thioxo-4,5-dihydro-1*H*-1,2,4-triazol-3-yl)-4,5-dihydropyridazin-3(2*H*)-one (**3**).

The purity of the compounds was checked by single-spot TLC, and the compounds were characterized on the basis of spectral data (IR, ¹H-NMR, MS) and elemental analysis. Spectral data of

the synthesized compound **3** was in full agreement with its proposed structure. The IR spectra revealed NH, CH, C=S, C=O and C=N absorption bands at 3323, 2930, 2368, 1685 and 1607 cm^{-1} , respectively. In the $^1\text{H-NMR}$ spectra, the signals of the respective protons of the title compound were verified on the basis of their chemical shifts, multiplicities, and coupling constants. The two triplets at δ 2.54 and 2.99 confirmed the presence of methylene protons at C-4 and C-5 position of the pyridazinone ring, respectively. The multiplet at δ 7.13–7.93 is due to the aromatic protons. The singlet at δ 10.78 is due to the hydrogen of the CSNH group. The $^{13}\text{C-NMR}$ spectrum showed peaks at δ 177 and δ 186 for a carbonyl carbon and a thioxo carbon. The mass spectrum shows the presence of a peak at m/z 350 ($M^+ + 1$) in accordance with the molecular formula. The elemental analysis results were within $\pm 0.4\%$ of the theoretical values. The starting material, 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide **1** was synthesized based on a literature method [15].

Figure 1. Synthetic route to the title compound **3**.



Synthesis of 6-phenyl-2-(4-phenyl-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-3-yl)-4,5-dihydropyridazin-3(2H)-one 3

An ethanolic solution of 6-oxo-3-phenyl-5,6-dihydropyridazine-1(4*H*)-carbohydrazide **1** (0.01 mol) and phenyl isothiocyanate (0.01 mol) was refluxed for 4 h. The contents were concentrated and poured into crushed ice, filtered and dried to give the crude thiosemicarbazide intermediate as 2-[(6-oxo-3-phenyl-5,6-dihydropyridazin-1(4*H*)-yl)carbonyl]-*N*-phenylhydrazinecarbothioamide **2**. The crude thiosemicarbazide intermediate **2** (0.005 mol) was refluxed in 2M sodium hydroxide solution (20 mL) for 5 h, cooled, poured into water with continuous stirring and filtered to give the final compound **3** as follows: the filtrate on acidification with glacial acetic acid yielded a solid which was recrystallized from ethanol. The purity of compound **3** was checked by TLC, using toluene/ethyl acetate/formic acid (5:4:1) as mobile phase and iodine (I_2) as visualizing agent.

Yield: 40%; m.p. 180–182 °C; R_f 0.42; white amorphous solid.

IR (KBr) ν_{max} (cm^{-1}): 3323 (NH), 2930 (CH), 2368 (C=S), 1685 (C=O), 1607 (C=N), 1030.

$^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 2.54 (t, $J = 8.7$ Hz, 2H, CH_2), 2.99 (t, $J = 8.7$ Hz, 2H, CH_2), 7.13–7.93 (m, 10H, Ar-H), 10.78 (s, 1H, CSNH).

^{13}C -NMR (75 MHz, CDCl_3): δ 27.1, 33.2, 124.5, 125.3, 128.6, 128.8, 129, 130.8, 131.2, 139.4, 154, 155, 155.6, 177 (C=O), 186 (C=S).

ESI-MS (m/z): 349/350 (M^+/M^++1).

Anal Calcd. for $\text{C}_{18}\text{H}_{15}\text{N}_5\text{OS}$: C: 61.87; H: 4.33; N: 20.04. Found: C: 61.82; H: 4.22; N: 19.96.

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