

Short Note

***N* -[(2,5-Dimethyl-3-oxo-1-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)carbamothioyl]isonicotinamide**

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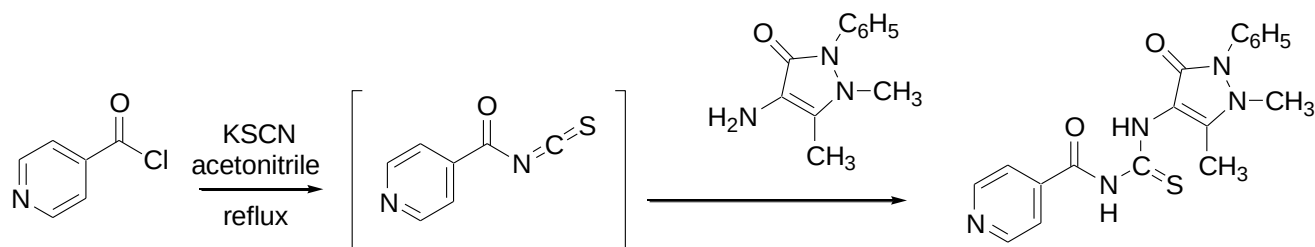
Abstract: A new heterocyclic compound, *N* -[(2,5-dimethyl-3-oxo-1-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)carbamothioyl]isonicotinamide, was synthesized from isonicotinoyl isothiocyanate and 4-aminoantipyrine in acetonitrile solution. The title compound was characterized by FT-IR, ¹H NMR, ¹³C NMR and MS spectroscopic methods. Physical parameters of the compound were examined.

Keywords: *N*-substituted thiourea; 4-aminoantipyrine; isonicotinoyl chloride

1. Introduction

N,N-Dialkyl-*N*'-acyl(aryl)thioureas, R/ArCONHC(S)NR'R', and *N*-alkyl-*N*'-acyl(aryl)thioureas, R/ArCONHC(S)NHR', have been known for more than 100 years and *N,N*-dialkyl-*N*'-arylthioureas were first synthesized by Neucki in 1873 [1,2]. These compounds, containing carbonyl and thiocarbonyl groups, have confirmed their utility among organic reagents as potential donor ligands for transition metal ions by different research groups [3,4]. Both thiourea derivatives and their metal complexes display a wide range of biological activities including antibacterial, antifungal, insecticidal, herbicidal, and plant-growth regulator properties [4–6]. In addition, pyrazole and its derivatives represent one of the most active classes of compounds; they possess a wide spectrum of biological activities, such as antibacterial, fungicidal, herbicidal and insecticidal activities [7].

In view of this, we report the convenient preparation of a new representative of this type of compounds.



2. Experimental

2.1. General

The ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE DPX NMR spectrometer operating at 400 and 101.6 MHz, respectively. Infrared absorption spectra were obtained on a Perkin Elmer BX II spectrometer from KBr discs, absorption bands are reported in cm^{-1} units. Carbon, nitrogen and hydrogen analyses were performed on a LECO CHNS-932 analyzer. Melting points were measured on an Electro Thermal IA 9100 apparatus using a capillary tube. LC mass spectra were obtained on an AGILENT 1100 MSD spectrometer with an ion source temperature of 240°C . 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydro-1H-pyrazol-3-one (4-aminoantipyrine), isonicotinoyl chloride hydrochloride, potassium thiocyanate, acetonitrile, and tetrahydrofuran were purchased from Merck.

2.2. *N*-[(2,5-dimethyl-3-oxo-1-phenyl-2,3-dihydro-1H-pyrazol-4-yl)carbamothioyl]isonicotinamide

A solution of isonicotinoyl chloride hydrochloride (0.356 g, 2 mmol) in acetonitrile (20 mL) was added dropwise to a suspension of potassium thiocyanate (0.196 g, 2 mmol) in acetonitrile (10 mL). The mixture was refluxed with stirring for 1 h. and then cooled to room temperature. A solution of 4-amino-1,5-dimethyl-2-phenyl-2,3-dihydro-1H-pyrazol-3-one (0.406 g, 2 mmol) in acetonitrile (20 mL) was added dropwise to the nicotinoyl isothiocyanate hydrochloride and stirred at ambient temperature for 4 h. The progress of the reaction was controlled by TLC. When the reaction was complete, the solution was poured into a beaker containing some ice. The yellowish precipitate was filtered off and washed several times with distilled water. After drying under vacuum, the material was recrystallized from tetrahydrofuran to give the product as yellow crystals; yield: 85%.

Melting point: $226\text{--}228^\circ\text{C}$

MS (EI): $m/z = 368.0$ ($[\text{M}+1]^+$)

IR (KBr, ν , cm^{-1}): 3101, 3061 (N-H), 2943 (C-H), 1721 (C=O), 1601 (C=O), 1496 (N-N), 1405 (N-CO), 1255 (N-CS), 1134 (C=S)

^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 12.04 (s, 1H, N-H), 11.38 (s, 1H, N-H), 8.80 (d, 2H, $^3J = 6.0$ Hz, pyridine-H), 7.86 (d, 2H, $^3J = 6.0$ Hz, pyridine-H), 7.37 (m, 3H, phenyl-H), 7.52 (m, 2H, phenyl-H), 3.33 (s, 3H, N-CH₃), 2.22 (s, 3H, C-CH₃)

^{13}C NMR (100MHz, $\text{DMSO}-d_6$): δ 182.18 (C=S), 167.47 (C=O), 161.46 (C=O), 153.33 and 108.71 ($\text{C}_{\text{pyrazole}}$), 150.63, 140.12, 135.44 ($\text{C}_{\text{pyridine}}$), 129.62, 124.32, 122.61 (C_{phenyl}), 38.14 (N-CH₃) and 11.93 (C-CH₃)

Elemental analysis: Calculated for $\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2\text{S}$: C, 58.84; H, 4.66; N, 19.06, S: 8.73, %. Found: C, 58.86; H, 4.63; N, 19.10, S: 8.71 %.

Acknowledgements

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References

1. Neucki, E. Zur Kenntniss des Sulfoharnstoffs. *Ber. Dtsch. Chem. Ges.* **1873**, 6, 598-600.
2. Douglass, I.B.; Dains, F.B. Some Derivatives of Benzoyl and Furoyl Isothiocyanates and their Use in Synthesizing Heterocyclic Compounds. *J. Am. Chem. Soc.* **1934**, 56, 719-721.
3. Koch, K.R. New Chemistry with Old Ligands: *N*-Alkyl- and *N,N*-Dialkyl-*N'*-acyl(aroyl) thioureas in Co-ordination, Analytical and Process Chemistry of the Platinum Group Metals. *Coordin. Chem. Rev.* **2001**, 216-217, 473-488.
4. Sandor, M.; Geistmann, F.; Schuster, M. An Anthracene-substituted Benzoylthiourea for the Selective Determination of Hg(II) in Micellar Media. *Anal. Chim. Acta* **1999**, 388, 19-26.
5. Del Campo, R.; Criado, J.J.; Garcia, E.; Hermosa, M.R.; Jimenez-Sanchez, A.; Manzano, J.L.; Monte, E.; Rodriguez, E.; Sanz, F. Thiourea Derivatives and Their Nickel(II) and Platinum(II) Complexes: Antifungal Activity. *J. Inorg. Biochem.* **2002**, 89, 74-82.
6. Xu, X.; Qian, X.; Li, Z.; Huang, Q.; Chen, G. Synthesis and Insecticidal Activity of New Substituted *N*-Aryl-*N'*-benzoylthiourea Compounds. *J. Fluorine Chem.* **2003**, 121, 51-54.
7. Bondock, S.; Rabie, R.; Etman, H.A.; Fadda, A.A. Synthesis and Antimicrobial Activity of Some New Heterocycles, Incorporating Antipyrine Moiety. *Eur. J. Med. Chem.* **2008**, 43, 2122-2129.

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