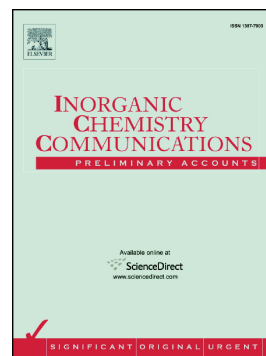


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Copper Complexes of New Thiosemicarbazone Ligands: Synthesis, Structural Studies and Antimicrobial Activity

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Abstract

Two thiosemicarbazone (TSC) ligands {HL¹ = 4-(2,5-dimethoxyphenyl)-1-((pyridin-2-yl)methylene)thiosemicarbazide and HL² = 4-(3-nitrophenyl)-1-((pyridin-2-yl)methylene)thiosemicarbazide} were prepared for the first time and characterized based on elemental analyses and spectroscopic (IR and electronic) measurements. Complexation reactions between the ligands and CuCl₂ resulted in the formation of two complexes {[CuCl(L¹)₂] **1** and [CuCl(L²)₂] **2**}. The crystal structure of **1** has been investigated by single crystal X-ray studies and was proved to be dimeric consisting of square pyramidal geometries around the Cu atoms as each Cu atom binds two bridging chlorine atoms and three atoms (two N, one S) from the TSC ligand. Ligands were tested against microbial strains and displayed no activity. Upon coordination, the activity was enhanced and, in some cases, was found comparable to that exhibited by the clotrimazole antifungal and chloramphenicol antibacterial standards.

Keywords: Thiosemicarbazones; Pyridine-2-carboxyldehyde Schiff bases; Coordination compounds; Crystal structure; Antimicrobial activity.

1. Introduction

Following their introduction, thiosemicarbazones (TSCs) have found growing interest from various research groups. Thanks to their diversity in many pharmaceutical and agrochemical applications[1-6]: these ligands and their metal complexes were proved to exhibit high biological activity against malarial[7], trypanosomel[8] and a number of fungal species[9] and displayed promising antitumor activity against various cancer cell lines[10-12]. The biological behavior of the TSCs enhances dramatically upon binding metal ions through a synergic effect[3,13,14]; it was proved that the TSC tendency to transport through biological membranes can be tuned by coordination.

Literature survey on Schiff bases of N(4)-thiosemicarbazones concluded that the biological behavior of this class of ligands depends on the nature of the carbonyl compound {aldehyde/ketone}, the existence of a bulky periphery at the terminal N4 nitrogen atom and the presence of a functionality as an additional binding site[15]. Nitrogen containing heterocyclic compounds represent vital part of many biologically active pharmaceuticals[16]. Schiff base tridentate ligands resulted from TSCs and pyridine-2-carboxyldehydes displayed significant biological activities[17-21] as they can interact with DNA and interfere in the DNA synthesis[22].

This article discusses the preparation of two new TSC ligands {HL¹ = 4-(2,5-dimethoxyphenyl)-1-((pyridin-2-yl)methylene)thiosemicarbazide and HL² = 4-(3-nitrophenyl)-1-((pyridin-2-yl)methylene)thiosemicarbazide} and their copper complexes. The article also describes the molecular structure of **1** elucidated by means of single crystal X-ray studies and the biological properties of the copper complexes and the free ligands against selected bacterial and fungal strains.

2. Experimental

a. Materials and physical measurements

The syntheses included high-purity analytical grade copper chloride and aryl isothiocyanates. Other chemicals and absolute solvents were purchased from commercial suppliers and were used as received. 4-(3-nitrophenyl)thiosemicarbazide[23] was prepared according to literature and 4-(2,5-dimethoxyphenyl)thiosemicarbazide was prepared following similar procedures.

Ligand melting points were determined in open capillaries and are reported uncorrected. CHNS analyses were conducted on an elemental Analysensysteme GmbH - vario EL III Element Analyzer. FT-IR spectra were recorded on a Nicolet iS10 spectrophotometer as KBr pellets in the range of 4000 - 400 cm⁻¹. Electronic spectral measurements were carried out over the wavelength range of 250-1000 nm on a Perkin-Elmer Lambda 40 UV/VIS Spectrometer. Room temperature magnetic moments were determined by using a Sherwood MKI magnetic susceptibility balance whereas Hg[Co(SCN)₄] was utilized as a calibration standard and the magnetic moments were corrected for diamagnetism by adding Pascal constants[24]. A Jenway 4320 conductivity meter was used to determine the molar conductivities of the complexes in DMF. PXRD studies for the complexes were determined on a Philips PW 1710 X-ray diffractometer equipped with nickel filtered CuK α radiation (λ = 1.54060 Å) and is operating at 40 kV and 40 mA in the 2 θ range of 4–90°.

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