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Synthesis and structural investigation of cu (II) complex of thiadiazole derivatives

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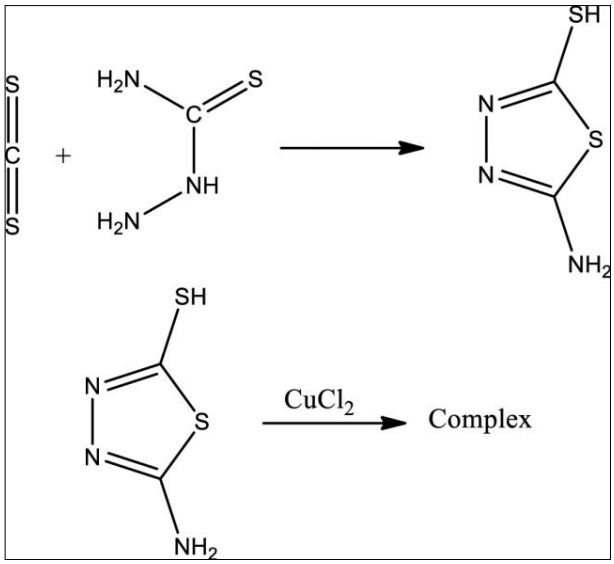
Abstract
A coordination complex of coinage metal Cu (II) with ligand 2,5-dimercapto-1,3,4-thiadiazole has been prepared. The complex was characterized using spectroscopic techniques including Ultraviolet-visible (UV–vis) and Infrared (FT-IR). The results obtained are consistent with the formation of dihydrate complexes, in which the chelation of the metal ion occurs via one of the thiadiazole nitrogen atoms and the deprotonated mercapto group of the aromatic ring. In the Cu (II) complex the ligand–metal ratio is 2:1.

Keywords: Mercapto group, 1, 3, 4-thiadiazole, thiadiazole complex

Introduction
A number of heterocyclic compounds such as imidazole, isothiazole, thiazole, Oxazole, pyrazole, pyrazine, quinoxaline, triazole, benzothiazole as well as many poly heterocyclic compounds are constituents of important physiological substances and biologically active organic compounds. These compounds have been widely used as donor molecules and their complexing behavior have been investigated in detail [1-5].
As the proposed work is concerned with the complexes of heterocyclic ligands containing nitrogen and sulphur as donor atoms, a short review of the earlier work done on the complexes of such ligands Is necessary to present here.

Experimental
In the first synthetic step, 2-amino-5-mercapto-1, 3, 4-thiadiazole will be prepared by the reaction of CS_2 with thiosemicarbazide was obtained as result of the classical $POCl_3$-mediated reaction between carbon disulphide and thiosemicarbazide. The compound 2-amino-5-mercapto-1, 3, 4- thiadiazole was then reacted with Cu (II) chloride salt.



Spectral Analysis

IR Spectroscopy

The IR spectrum of the ligand and its Cu (II) complex with the ligand 2-amino-5-mercapto-1, 3, 4- thiadiazole has been investigated. The high frequency region in the FT-IR spectrum of the ligand is dominated by three sharp bands at 3385, 3320, and 3206 cm^{-1}. Intramolecular hydrogen bonding between the thiadiazole nitrogen and the thiole group residing at carbon C2 results in low intensity and significant broadening of the -SH stretching band [6]. The two sharp bands in this region, namely at 3320 and 3206 cm^{-1}, represent the respective symmetrical and asymmetrical stretches of the -NH2 group [7]. The thiadiazole ring formation manifests in the presence of a strong and sharp band at approximately 1630cm^{-1} characteristic of the heterocyclic -C=N- stretches [8]. Another sharp band at 1600 cm^{-1} represents the N-H bending vibrations of the amine [9]. The central part of fingerprint region (1320–1120 cm^{-1}) is occupied by a series of sharp bands attributed to the in-plane N-H bending and the C-N stretching vibrations. A series of weak intensity bands below 660 cm^{-1} originates from the -C-S-C- stretching of the thiadiazole ring as well as out-of-plane vibrations of the hydroxyl S-H bonds [10].

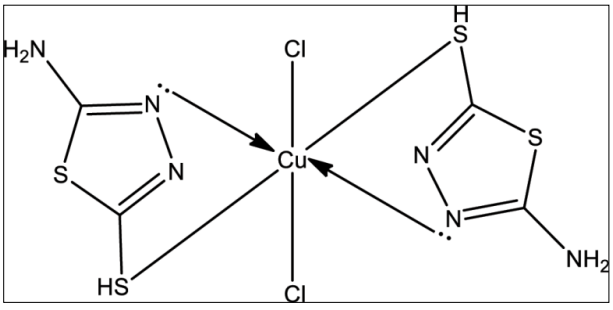
The most significant change in the central part of the fingerprint region is due to the appearance of sharp band at 974 cm^{-1}, which may be assigned to the N-H out-of-plane bending vibration. The second most significant change in this region is a band at 623 cm^{-1} suggesting the structural alterations made near the -C-S-C- system of the thiadiazole ring [11].

The spectra of complex revealed a broad and moderate intensity bands spanning from approximately 3500 to 3000 cm^{-1} suggesting the presence of hydrates, consistent with microanalysis result. This broad band overlapped the remaining characteristic bands which are normally expected to appear in this region, and particularly the N-H stretching. Thus, the involvement of the -NH^2 group in the complex formation cannot be confirmed by IR spectroscopy.

Compared to the spectrum of free ligand, the corresponding complex of Cu (II) revealed [12] a moderate shift of the thiadiazole C=N band, from 1628 cm^{-1} to 1607. This points at the ligand–metal interaction occurring via thiadiazole ring nitrogen and is further evidenced by numerous changes in the fingerprint region of the spectra, suggesting the metal chelating by both thiadiazole nitrogen and the nearby deprotonated -SH group.

In addition, another band appears at ~725 cm^{-1} region in the complex indicating the complexation of chloride ion through metal (Cu^{2+}) ions [13].

On the basis of the above discussion, the structure of the complex may be shown as:



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