DESIGN AND SYNTHSIS OF ZINC (II) COMPLEXES WITH SCHIFF BASE DERIVED FROM SULPHAGUANIDINE AND HETEROCYCLIC ALDEHYDES

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ABSTRACT

A unique series of Zinc (II) complexes were synthesized by the reaction of a Zinc (II) acetate with a Schiff base created by the condensation of 2-acetyl thiophene or 2-acetyl furan with 2-(4-aminophenyl) sulfonylguanidine (Sulphaguanidine) in a 1:1 molar ratio. The coordination behaviour of Schiff bases towards Zinc atoms has been investigated using a variety of techniques, including elemental studies, conductance measurements, molecular weight estimations, and IR, UV, and 1H NMR spectrum examinations.

KEYWORDS : Schiff base, Zinc (II) acetate, 2-Acetyl Thiophene, 2-Acetyl Furan, Condensation, Metal complexes, Sulphaguanidine, Sulpha drugs, IR, UV, Elemental analysis, NMR.

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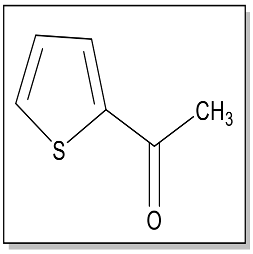
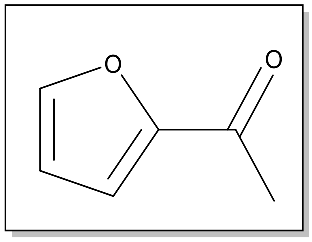
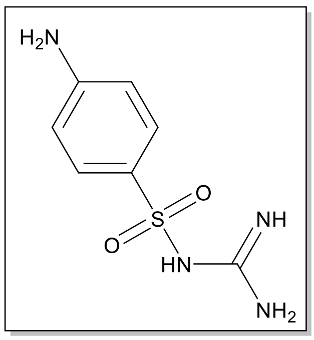
INTRODUCTION

The sulfonamides are a significant class of medications, with many pharmacological agents exhibiting, among other properties, anticancer[1], antibacterial[2], anti-carbonic anhydrase[3,4], diuretic[3,5], antithyroid[6] , hypoglycemia[7], or protease inhibitory activity[8-10].The sulfamoyl functional group is connected to aniline at the 4-position in sulfanilamide, a sulfonamide. It functions as an antibacterial agent, an EC 4.2.1.1 (carbonic anhydrase) inhibitor, and a drug allergen. It is a sulfonamide antibiotic, a substituted aniline, and a sulfonamide. Sulfaguanidine is a sulfonamide that has a guanidine moiety that is mostly used in veterinary medicine to prevent the formation of folic acid. It serves as an anti-infective agent. a sulfanilamide antibacterial medication used to treat intestinal infections.It was, therefore, considered of interest to synthesize Zn(II) derivatives of Schiff bases derived by the condensation reaction of 2-Acetyl Thiophene and 2-Acetyl Furan with Sulphaguanidine. In this paper, we are communicating the results of these studies. The structures of the starting materials are shown in fig. 1.







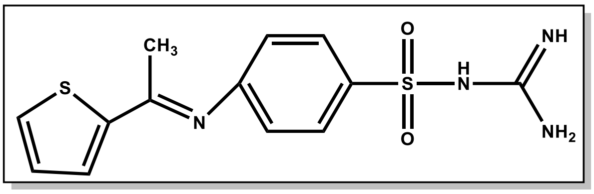
2-Acetyl Thiophene 2-Acetyl Furan
Sulphaguanidine

METHODOLOGY

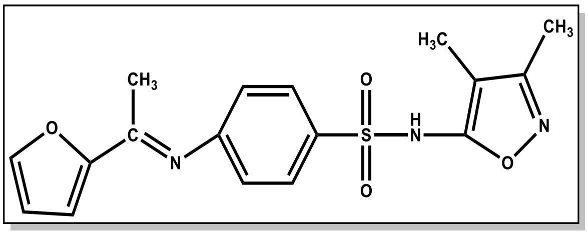
All the chemicals used in this research are of AR grade, standard protocols were followed for drying the solvents and anhydrous and oxygen-free atmosphere was maintained while carrying out all the reactions.

Synthesis of ligands

Sulphaguanidine and 2-Acetylthiophene/2-Acetyl Furan were condensed in a 1:1 molar ratio to yield the Schiff base, which was then refluxed using ethanol as the reaction medium for six hours on a water bath. Following the reflux, the schiff base crystals were separated on cooling using filter paper, and the crystals were then washed with ethanol. They were dried, and acetone was used to recrystallize them. Schiff base ligands' structures are shown in fig.2. Table 1 lists the key characteristics and analyses of these ligands.



2-Acetylthiophene Sulphaguanidine



2-Acetylfuran Sulphaguanidine

Synthesis of Zinc (II) Complexes

Then, using methanol as the reaction medium, the ligands were reacted with zinc (II) acetate at a molar ratio of 1:2. The reaction was then refluxed for 8 hours, with the progress of the reaction being monitored by measuring the amount of acetic acid in the azeoptrope. The products were cleaned with dry cyclohexane after the solvent was removed, and they were then dried in vacuo. The major traits and analyses of these complexes are listed in Table 2.The reaction of zinc (II) acetate with the ligand and methanol in 1:2 molar ratio can be represented as:

$$(AcO)_2Zn.4H_2O + 2N\sim NH + MeOH \longrightarrow [Zn(N\sim N)_2.2H_2O] + 2AcOH + 2H_2O$$

Where N~NH represents the bi-functional tridentate Schiff base ligand. The resulting complexes are coloured solids (Table 2), which are soluble in DMSO, alcohol, benzene, carbon tetrachloride, chloroform and DMF.At ambient temperature, these complexes act as non-electrolytes in DMF, with molar conductance values ranging from 12 to 18 $ohm^{-1}cm^2mol^{-1}$. The magnetic moment values at ambient temperature (303K) demonstrate the diamagnetic nature (0.04-0.20 B.M.).

RESULTS AND DISCUSSION

IR Spectra

Due to the free azomethine group's υ(C=N) frequency emerging in the ligands, a noticeable and strong band is seen in the region of 1670–1710 cm^{-1} in the enhanced spectra of the ligands. In the spectra of metal complexes, this band is shifted to the lower region (~15 cm^{-1}), demonstrating the coordination of the metal atom with the nitrogen of the azomethine. This is further supported by the presence of (Zn→N) at 1350 cm^{-1} in the spectra of zinc metal complexes.

A broadband in sulpha drugs is seen between 3355 and 3315 cm^{-1} and is ascribed to (N-H), although it does not appear in complexes that indicate this group's deprotonation. The presence of an additional band at 875 cm^{-1} in the spectra of complexes of the type $[Fe(NN)_2.2H_2O]$ suggests the presence of a coordinated water molecule.

^{1}H NMR Spectra

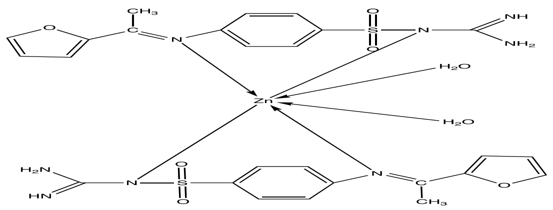
The ligands' zinc (II) complexes' ^{1}H NMR spectra in DMSO-d_6 were recorded. According to the methyl proton signal at δ~1.50 ppm, which shifts at δ~1.60±0.02 ppm in the spectra of metal complexes, coordination to the metal atom may occur through the nitrogen of the azomethine. The schiff bases' δ~12.56ppm, which is attributable to the -OH proton that does not exist in the metal complexes, may act as a bond between the oxygen atom and the metal. Furthermore, in metal complexes, a signal at δ~5.0 ppm attributed to the methine group in the schiff base changes to δ~1.5±0.02 ppm, demonstrating the existence of a coordination connection between (N-Zn).

A signal at δ~10.98 ppm appears in Schiff bases of sulpha medicines but not in metal complexes, indicating that the nitrogen atom has been chelated to the metal atom.

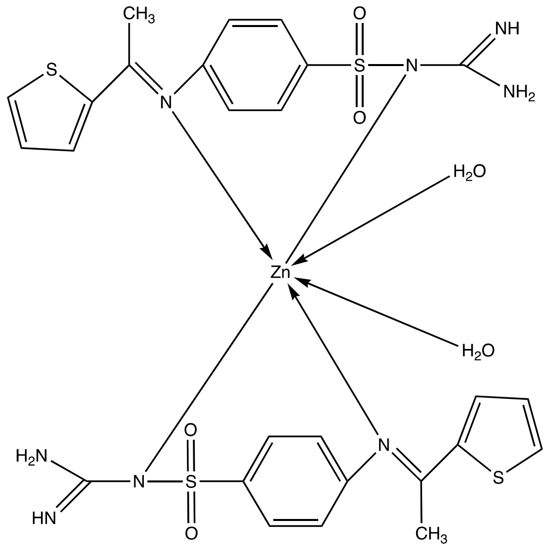
Electronic Spectra

Two bands in the ligands' electronic spectra, which are characteristic of the spectrum of sulpha medicines and are linked to the ϕ-ϕ* and π-π* benzenoid transition, are located around 255 nm and 310 nm. Due to the azomethine group's n-π* electronic transition, a new band with a wavelength of 415 nm is seen in the resulting zinc (II) complexes.

On basis of the spectral data and elemental analysis the following structure can be assigned to these newly synthesized derivatives as shown below:



2-Acetylfuran Sulphaguanidine Zinc Complex



2-AcetylThiophene Sulphaguanidine Zinc Complex

Measurements

Kjeldahl's technique was used to calculate nitrogen. The complexes were examined as previously stated[17]. KBr discs were used to record infrared spectra on a Perkin-Elmer-577 spectrophotometer in the 400-200 cm-1 range. The PMR spectra were obtained using a Perkin-Elmer Model RB-12 spectrometer with DMSO-d_6 as the solvent and TMS as the internal standard. A systronics conductivity bridge model was used to quantify molar conductance in anhydrous DMF at 36°C. The Rast camphor technique was used to determine the molecular weights.

Sr. No.	Ligands	Colour	M.P.(C)	Elemental Analysis %					
				C Found (Calcd)	H Found (Calcd)	N Found (Calcd)	O Found (Calcd)	S Found (Calcd)	Mol. Wt. Found (Calcd)
1	2-Acetylthiophene sulphaguanidine ($C_{13}H_{14}N_4O_2S_2$)	Creamish white solid	108	48.28 (48.43)	4.21 (4.38)	17.28 (17.38)	9.71 (9.92)	19.12 (19.86)	310.42 (328.11)
2	2-Acetylfuran sulphaguanidine ($C_{13}H_{14}N_4O_3S$)	Cream solid	130-134	50.84 (50.96)	4.51 (4.61)	18.12 (18.29)	15.34 (15.67)	10.12 (10.45)	301.41 (306.11)

Table 1: Elemental Analysis Of Sulpha Ligands

Sr No	Reactants Zinc Compound (g)	Ligands (g)	Molar Ratio	Product (Colour & State)	M.P. (C)	Elemental Analysis %						Mol. Wt. Found (Calcd)
						C Found (Calcd)	H Found (Calcd)	N Found (Calcd)	O Found (Calcd)	S Found (Calcd)	Zn Found (Calcd)	
1	$Zn(OOCCH_3)_2$	2-Acetylthiophene sulphaguanidine ($C_{13}H_{14}N_4O_2S_2$)	1:2	$C_{26}H_{26}N_8O_4S_4Zn$ Light Yellow solid	148	43.80 (44.10)	3.20 (3.70)	15.30 (15.82)	9.00 (9.04)	18.03 (18.11)	9.04 (9.23)	703.57 (708.66)
2	$Zn(OOCCH_3)_2$	2-Acetylfuran sulphaguanidine ($C_{13}H_{14}N_4O_3S$)	1:2	$C_{26}H_{26}N_8O_6S_2Zn$ Yellow Solid	169	46.05 (46.19)	3.31 (3.88)	16.12 (16.58)	14.03 (14.20)	9.31 (9.48)	9.52 (9.69)	670.11 (676.04)

Table 2: Elemental analysis of Zinc (II) complexes

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