



Research Article

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## Synthesis and characterization of Schiff base complexes of Cu[II] and Co[II]

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### ABSTRACT

The complexes [SGC, SGCO] of Cu [II], and Co [II] ions with a Schiff base [SG] derived from salicylaldehyde and sulfaguanidine have been investigated. The resulting complexes were characterized by UV/Vis IR, <sup>1</sup>HNMR spectral studies, elemental analysis, molar conductance, XRD, Magnetic susceptibility and antimicrobial activity. The molar conductance measurement indicates that the complexes were non electrolytes. The IR data shows that complexes were mono basic ligand with NO bidentate sites. The XRD study shows that the complexes were microcrystalline in nature, the antimicrobial activity of the complexes were tested against the bacterial, *E.coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus* and fungi *Candida* by the Disc diffusion method. The complex shows stronger antimicrobial activity than the free ligands.

**Key words:** Schiff base, Sulfaguanidine, Antifungal and Antibacterial activities.

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### INTRODUCTION

The study of Schiff base complexes of metals were eternal and drew the attention of scientists of all time due to a variety of applications in biological, clinical, analytical and pharmacological areas[1-3]. Tetra dentate Schiff bases with N<sub>2</sub>O<sub>3</sub> donor atom set are well known to coordinate with various metal ions and this had attracted the interest of many authors [4-15]. Complexes of transition metals [II] which involve derivatives of salicylaldehyde and sulfa drugs have received considerable attention. Transition metal Schiff base complexes were used in various fields such as medicine, agriculture, and industries. Sulfa drugs were an important class of therapeutic compounds. In combination with certain other drugs, they were used for the treatment of various bacterial infections. Studies of the coordination behavior of sulfadugs were of considerable interest because the coordination of metal ion was reported to enhance the biological activities of organic compounds. The schiffbases derived by the condensation of sulfonamides with salicylaldehyde were not only good complexing agents but also good bacteriocides. Chelating ligand containing O and N donor atoms show broad biological activity and special interest of variety of ways in which they were bonded to metal ions [16]. This paper reports the studies of Cu [II], Co [II] complexes of Schiff base derived from salicylaldehyde and sulfaguanidine due to paucity of information.

### EXPERIMENTAL SECTION

Salicylaldehyde [Fluka], Sulfa guanidine [Fluka], Ethanol was used without further purification. Copper [II] nitrate and Cobalt perchlorate were reagent grade. A Fisher-100 infrared spectrophotometer was used to record the IR spectra as KBR and CSI disc at NIIST Trivandrum. A UV/Vis spectrum was measured by a Double Beam Spectrometer-2203. <sup>1</sup>HNMR Spectra of the synthesized compounds were recorded at NIIST Trivandrum using DMSO solvent and TMS as the internal standard. The Elemental analysis [C,H,N,S] were carried out using micro analytical technique on C,H,N,S,O using Elementa analyser at CUSAT Cochin. Molar conductivity measurements were recorded on a CM-82T Elico conductivity bridge in DMSO. The antifungal and antibacterial activities were

tested by the diluted samples were plated on to Gelatin agar plates incubated at 37°C for 24 hours by Disc diffusion method.

### Synthesis of Schiff base

Added Salicylaldehyde [0.06ml, 0.1mmol] in 10ml absolute ethanol dropwise stirring to sulfa guanidine [0.107mg, 0.1mmol] in 10ml of ethanol in a beaker with the molar ratio 1:1. The reaction mixture was heated for 30 minutes during which the colour of the solution change to yellow the yellow solid product was filtered and recrystallized in ethanol after 5hours.

### Synthesis of complexes

A solution of 20ml copper nitrate [0.1mmol]/Cobalt per Chlorate [0.1mmol] in ethanol and added to a solution of ligand of sulfa guanidine. The reaction mixture was heated for 22 hours the complex precipitate was separated by filtration .Washed with ethanol and dried.

## RESULTS AND DISCUSSION

All the complexes were insoluble in water, ethanol and methanol but soluble in DMSO.

### IR spectra

The IR spectral data of the ligand showed a band at 1618cm<sup>-1</sup> which was assigned to  $\nu$  [CH=N] stretching vibration a feature found in Schiff bases. This band is observable in the complex compounds suggesting that the ligand has coordinated to the respective metal ions resulting the formation of the complexes. The bands in the region 511-540cm<sup>-1</sup> and 450-500cm<sup>-1</sup> are attributed to [M-O] and [M-N] stretching vibrations respectively. The broad band in the region 3350-3560cm<sup>-1</sup> was accorded to  $\nu$  [O-H] stretching vibrations indicating the presence of water. The bands 1322cm<sup>-1</sup> and 1152cm<sup>-1</sup> were not changed because sulfonamide O<sub>2</sub> were not contributing in coordination with the metal ions. The IR data of ligand and their complexes were presented in table1.

Table 1 The IR spectra of the Schiff base and the complex

Compound	$\nu$ [C=N]	$\nu$ [O-H]	$\nu$ [SO <sub>2</sub> ]as	$\nu$ [SO <sub>2</sub> ]s	$\nu$ [C=O]	$\nu$ [M-O]	$\nu$ [M-N]
Schiff base	1618	3327	1321	1159	1569	511	450
SGC	1632	3226	1321	1159	1539	514	500
SGCO	1620	3456	1322	1152	1529	540	522

SGC – Copper complex, SGCO – Cobalt Complex, SG – Schiff base

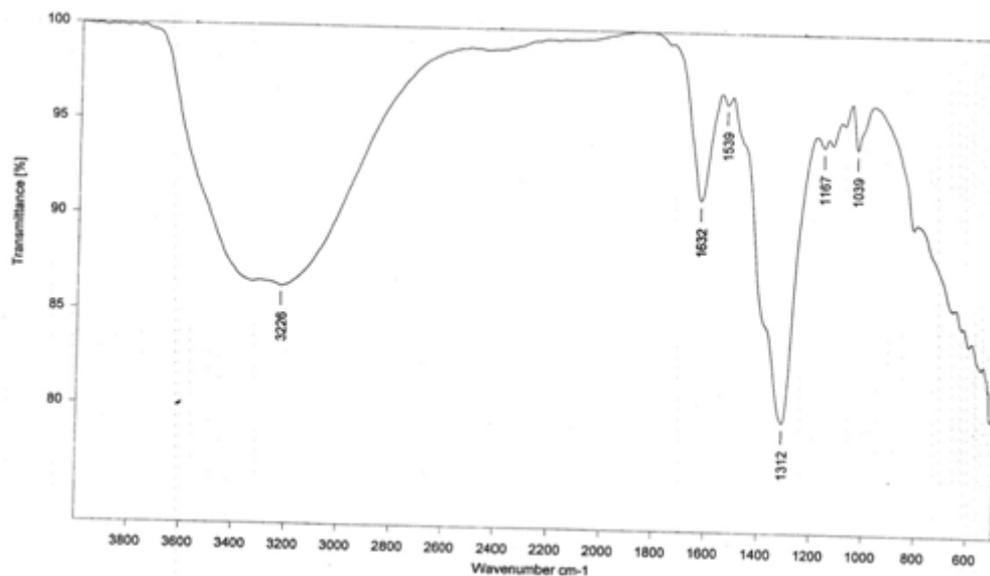


Fig:1 IR Spectra of SGC

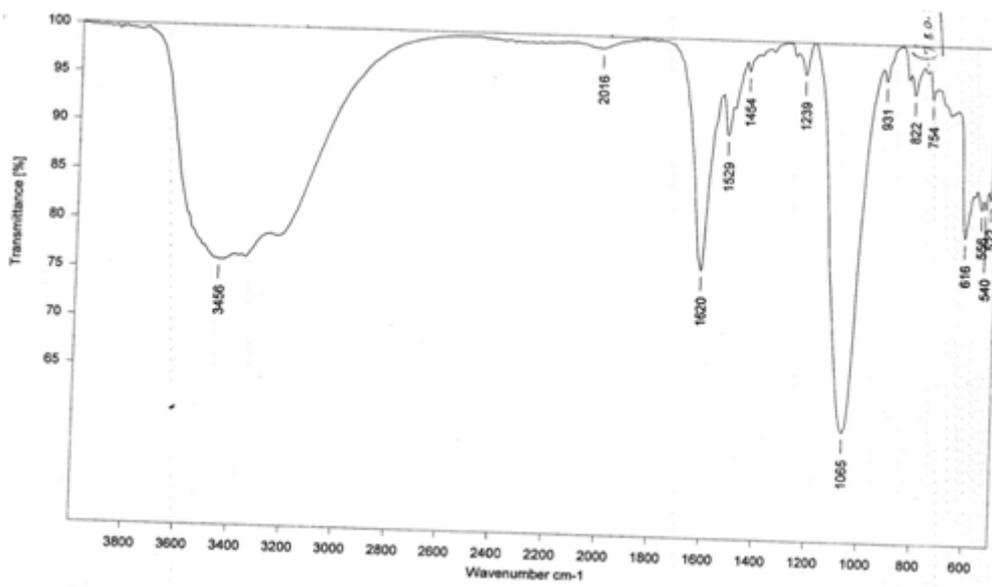


Fig:2 IR Spectra of SGCO

### <sup>1</sup>HNMR spectra

In the <sup>1</sup>HNMR Spectrum of the schiffbase SGC, SGCO recorded the characteristic singlet at 8.23ppm is due to the azomethine proton [-CH=N]. The signal at [2.31] is assigned to methyl group. The azomethine [CH=N] proton which was shifted to downfield in the spectra of all the complexes. This is attributed to the donation of lone pair of electrons by the azomethine nitrogen to the metal atom. The-OH proton signals were formed 10.40-11.80ppm. This was indicating the involvement of phenolic O<sub>2</sub> in the coordination of complexes. The <sup>1</sup>HNMR spectra of ligands in DMSO-d<sub>6</sub> revealed a multiplet at 6.01-7.95ppm corresponding to aromatic protons. Thus the NMR results support the IR inferences.

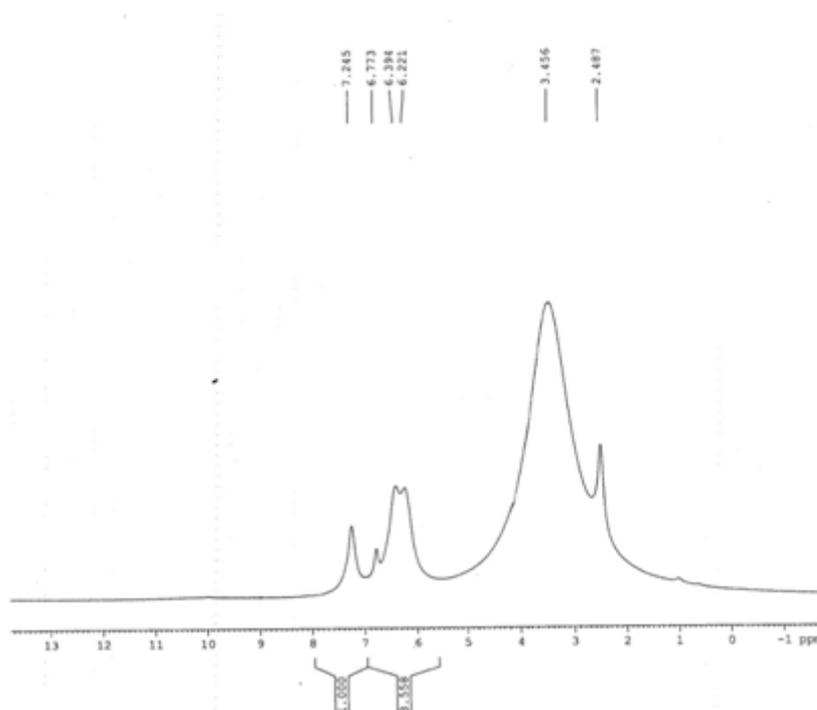


Fig 3: NMR Spectra of SGC

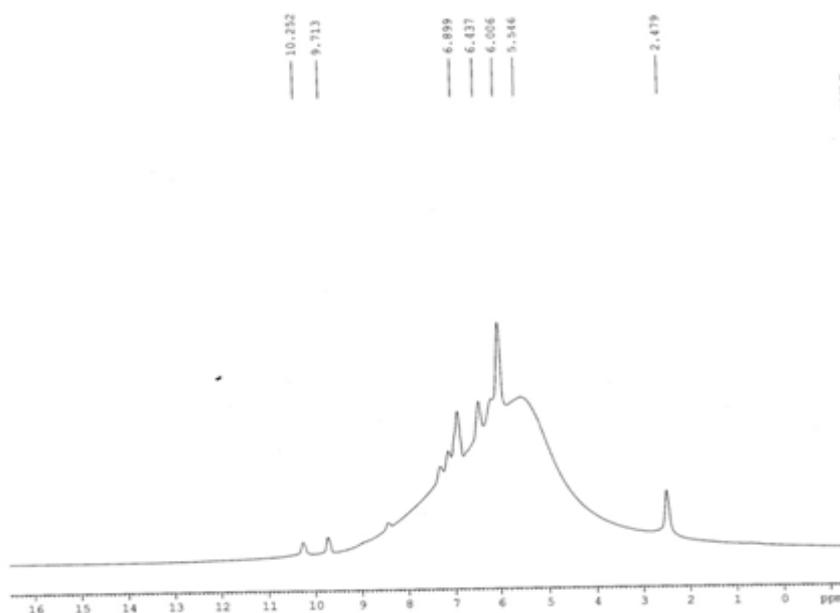


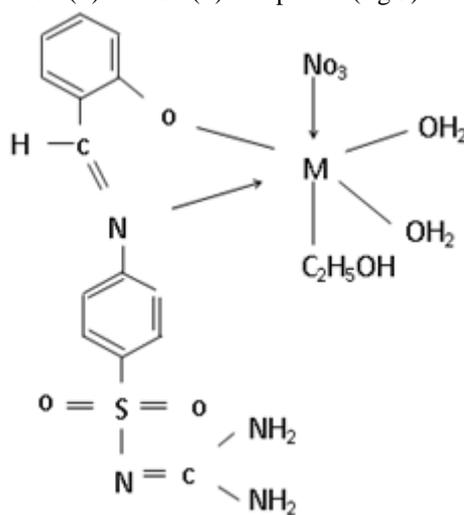
Fig 4: NMR Spectra of SGCO

Table 2: Analytical Data of the ligand and its complexes

S.No	Complexes	Color	Magnetic moment	Elemental Analysis % [found/ calculated]					
				C	H	N	O	S	M
1	SG[C <sub>14</sub> H <sub>14</sub> N <sub>4</sub> O <sub>3</sub> S]	Yellow		52.8	4.40	17.6	15.09	10.06	--
				49.95	4.16	16.65	14.27	9.51	
2	SGC[C <sub>15</sub> H <sub>25</sub> N <sub>5</sub> O <sub>9</sub> SCu]	Dark brown	1.67	34.98	4.8	13.36	27.49	6.10	12.34
				34.36	4.7	13.60	27.98	6.29	12.13
3	SGCO[C <sub>15</sub> H <sub>25</sub> N <sub>5</sub> O <sub>9</sub> SCo]	Orange	3.73	35.29	4.9	13.72	28.23	6.27	11.55
				34.66	4.8	13.48	27.73	6.16	11.34

### Magnetic susceptibility

The magnetic susceptibility was measured by Guoy balance method. The observed  $\mu_{\text{eff}}$  values present in the table to suggest that the octahedral geometry of Cu (II) and Co (II) complexes (fig 5).



M=Cu or Co  
Fig 5: Structure

### Conductance measurements

The observed molar conductance was  $6.9\text{-}12.0\text{ Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$  all the complexes in  $10^{-3}$  molar solution in DMSO were suggest the non-electrolytic nature of these complexes. Such a non zero molar conductance value for each of the complex in the present study is most probably due to the strong donor capacity of DMSO which may lead to the displacement of anionic ligand and charge of electrolytic type [17].

### UV spectra

The electronic spectra of the ligand and the complexes were recorded DMSO solvent in the UV visible region. An absorption band found at the electronic spectra of the metal complexes recorded in UV region exhibit intra ligand and charge transfer transition of the C=N chromophore. On complexation this band was shifted to lower wavelength region at 250nm and 249nm suggesting the coordination of azomethine nitrogen with Cu [II] and Co [II] ions due to  $\pi\text{-}\pi^*$ . The spectra also shows the other transitions in the range of 246nm and 247nm which can be assigned to  $n\text{-}\pi^*$  transitions. Transitions around 600-650nm may be due to d-d transitions which are characteristic feature of transition metal complexes. In the complexes bands below 300nm were attributed to intra ligand transitions [18-19].

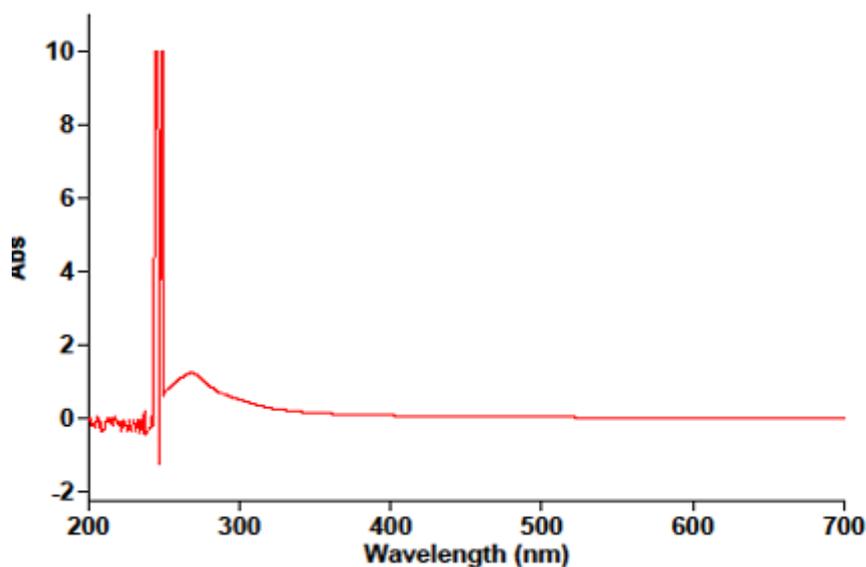


Fig 6: UV Spectra of SGC

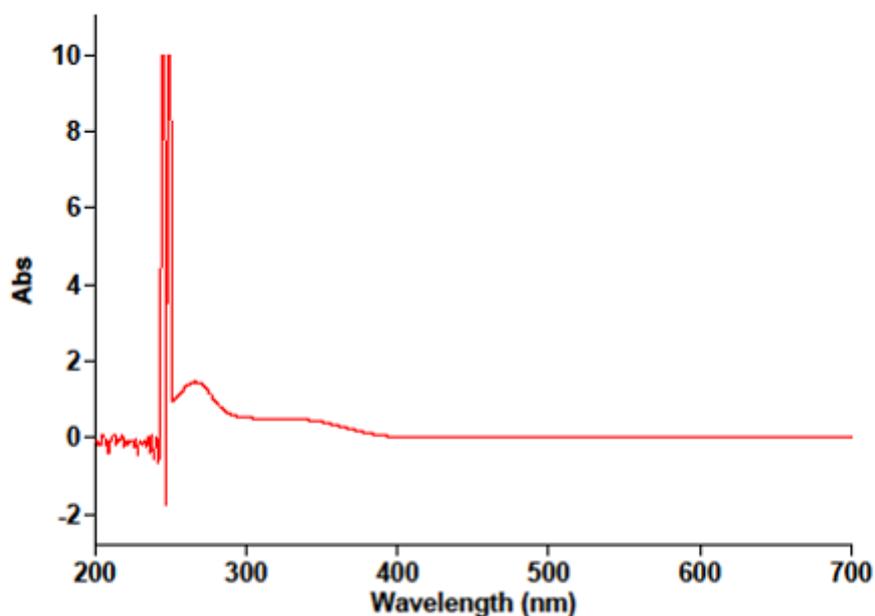


Fig 7: UV Spectra of SGCO

**XRD**

The crystalline structure was performed by X ray diffraction analysis [XRD] utilizing Cu-K $\alpha$  [wavelength 1.5406 Å] radiation. Using the Scherer formula the crystallite size of the prepared complexes of SGC, SGCO have been calculated. Scherer formula can be written as [19].

$$\text{Crystallite size } D = 0.94\lambda / \beta \cos\theta$$

$\lambda$  is the wavelength of x ray used

$\beta$  is the full width at half maximum

$\theta$  is the diffraction angle

Using this formula the size of the ligand complexes was microcrystalline in nature.

**Biological activity**

Antifungal and anti bacterial activities of the sulfaguanidine and its copper complex and cobalt metal complexes were studied against E.coli Pseudomonas aeruginosa Staphylococcus aureus and Candida in Agar media, the petri dishes were incubated at 24 hours at 37<sup>o</sup>c. The results were indicated that in the table 3.

**Table 3: Zone of Inhibition (mm)**

S.No	Organisms	Media	Chloramphenicol	SG	SGC	SGCO	NC
1	<i>E.coli</i>	Muller Hinton Agar	23.0	6.0	6.0	19.0	6.0
2	<i>Pseudomonas aeruginosa</i>		21.0	6.0	6.0	6.0	6.0
3	<i>S. aureus</i>		22.0	6.0	6.0	6.0	6.0
4	<i>Candida</i>		22.0	6.0	6.0	6.0	6.0

**CONCLUSION**

The spectral data show that the ligand act as bidentate coordinating through nitrogen atom of the azomethine and oxygen atom of hydroxyl group of Salicylaldehyde. The XRD data shows that the ligand complexes were microcrystalline in nature. The molar conductance shows that the ligand complexes were non electrolytic in nature. The SGC complex was least active and the complex of SGCO was highly active in E.coli. This effect may be due to the coordination of metal ion. They killed the cells of bacteria and fungi. So they can be used as anti bacterial and antifungal agents.

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