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# Synthesis and characterization of Schiff base Guanidine complexes of Ni[ll]

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**ABSTRACT**

The complexes [SGN] of Ni [ll] ion with a Schiff base [SG] derived from salicylaldehyde and sulfaguanidine have been investigated. The resulting complexes were characterized by UV/Vis IR, 1HNMR spectral studies, elemental analysis, molar conductanceand XRD, . 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.

**Keywords:** Schiff base, Sulfaguanidine, XRD.

## 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 N2O3 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 [ll] 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 sulfadrugs 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 [ll], Co [ll] complexes of Schiff base derived from salicylaldehyde and sulfaguanidine due to pacity of information.

## EXPERIMENTALSECTION

Salicylaldehyde [Fluka], Sulfa guanidine [Fluka], Ethanol was used without further purification. Ni [ll] nitrate was 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. 1HNMR 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.

## 2 EXPERIMENTAL METHODS

### 2.1 Synthesis of Schiff base of Salicyl sulphaguanidine[SG]

Added Salicylaldehyde[0.06ml,0.1m mol]in 10ml absolute ethanol drop wise stirring to sulpha guanidine[0.107mg,0.1mmol] in10ml of ethanol in a beaker with molar ratio1:1. The reaction mixture was heated to50$^0$C over a water bath.Formation of the shiff base was indicated by the development of yellow colour. The yellow solid product was filtered and crystallized in ethanol after 5 hours. The melting point was 241$^0$C.

### 2.2 Synthesis of Salicyl Sulpha drug Schiff base Nickel[II] complexes

A aqueous solution of Nickel nitrate was added dropwise to the schiff bases salicylsulphaguanidine] with constant stirring. The mixture was kept at 50C with constant stirring.and heating for 22-28 hours. The solid product was collected on a filter, washed successively with water ,alcohol and finally with ether. The product was dried in vaccum over fused calcium chloride

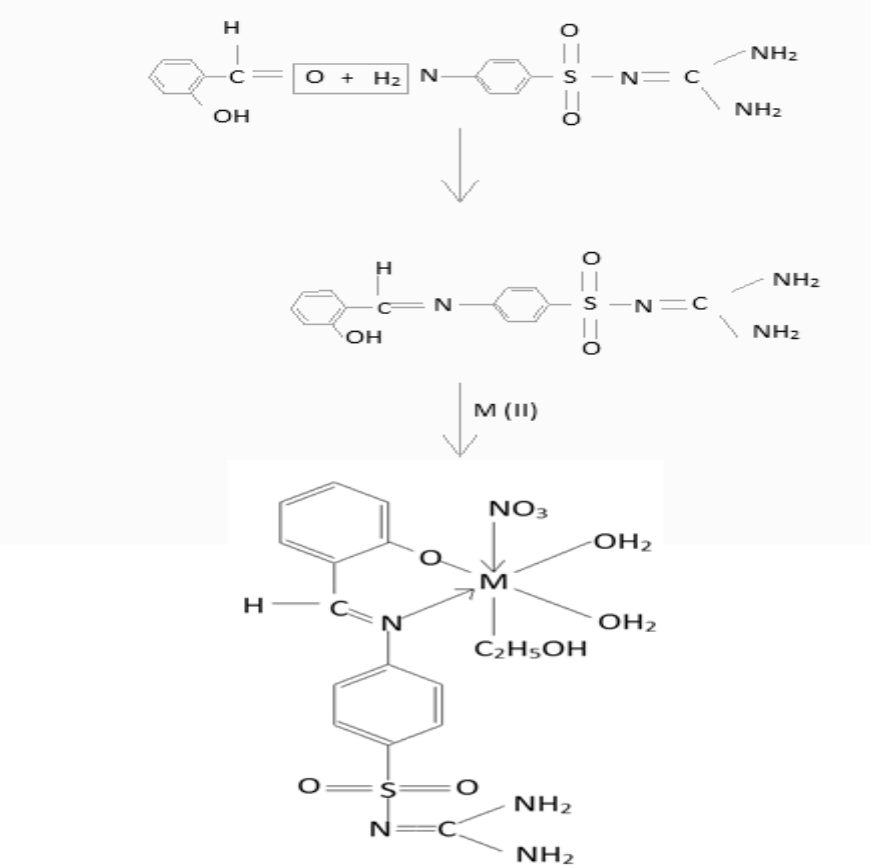


**Figure 1: Synthesis of salycyl sulphaguanidine Schiff base metal[II] complexes**

### 2.3 ANALYTICAL STUDIES

#### 2.3.1 Elemental Analysis

The elemental analysis C,H,N,S,O were carried out using elemental analyzer at CUSAT Cochin. In the analysis 10 mg sample weighing concealed in a tin capsule was injected in the instrument and the compu-

terized elemental percentage was obtained EDTA was used for calibrating the instrument.

**2.3.2 IR spectra**

The IR spectra were recorded as KBr pellets in the region 400-4000cm$^{-1}$ using a Fisher 100 spectrophotometer was used to record the spectra as CSI disc at NIIST Trivandrum.

**2.3.3 UV/Visible Spectra**

A UV/Vis spectrum was measured by Schimaduz Double Beam visible spectrometer using a quartz cell 1cm optical path; The electronic spectral measurements were carried out to the Schiff base and their metal complexes of Ni in DMSO were used to determine the geometry of complexes-

**2.3.4 $^{1}$HNMR Spectra**

$^{1}$HNMR spectra of the synthesized compounds were recorded at NIIST Trivandrum using DMSO solvent and TMS as the internal standard. $^{1}$HNMR spectra were recorded on a perkin Elemer RB12spectrometer in $CDCl_3$ using TMS an internal standard at 90MHZ. The $^{1}$HNMR spectra give information about the electronic structures of the complexes;

**2.3.5 Molar conductivity measurements**

Conductance measurements are useful in determining the electrolytic nature of the complexes. The molar conductivity measurements were recorded on CM-82T Elico Conductivity Bridge in DMSO. The molar conductivity of the complexes were determined using the formula

$$X_m = \frac{1000 \times conductance \times cell\ constant}{concentration}$$

**2.3.6 Powder XRD**

The powders XRD were recorded using SHIMADUU model XRD6000 instrument with Cuk radiation1.54060 at NIIST Trivandrum. X-ray diffraction technique used for structure determination and crystalline size of complexes.

## 3. RESULTS AND DISCUSSIONS

### 3.1. IRSALICYL SULPHA GUANIDINE[SG] AND ITS COMPLEXES

The IR spectral data of the schiff base ligand showed a band at1618cm$^{-1}$ which was assigned to ν[CH=N] stretching vibration a feature found in Schiff base SG. This indicates the participation of the nitrogen atom of the azomethine group in coordination. This band is shift in the complex compound SGN suggesting that the ligand has coordinated to the respective metal ions resulting the formation of the complexes. The bands in the region 523cm$^{-1}$-555cm$^{-1}$ and 500cm$^{-1}$-522cm$^{-1}$ are attributed to [M-O] and [M-N] stretching vibrations respectively. The broad band in the region 3145-3456cm$^{-1}$ was accorded to ν[O-H] stretching vibrations indicating the presence of water 1524-1580cm$^{-1}$. The bands 1321cm$^{-1}$, 1159cm$^{-1}$ were not changed because sulfonamide oxygen were not involved in coordination with the metal ions. The IR data of shiff base ligand and their complexes were presented in table 1.and also in figure 2.

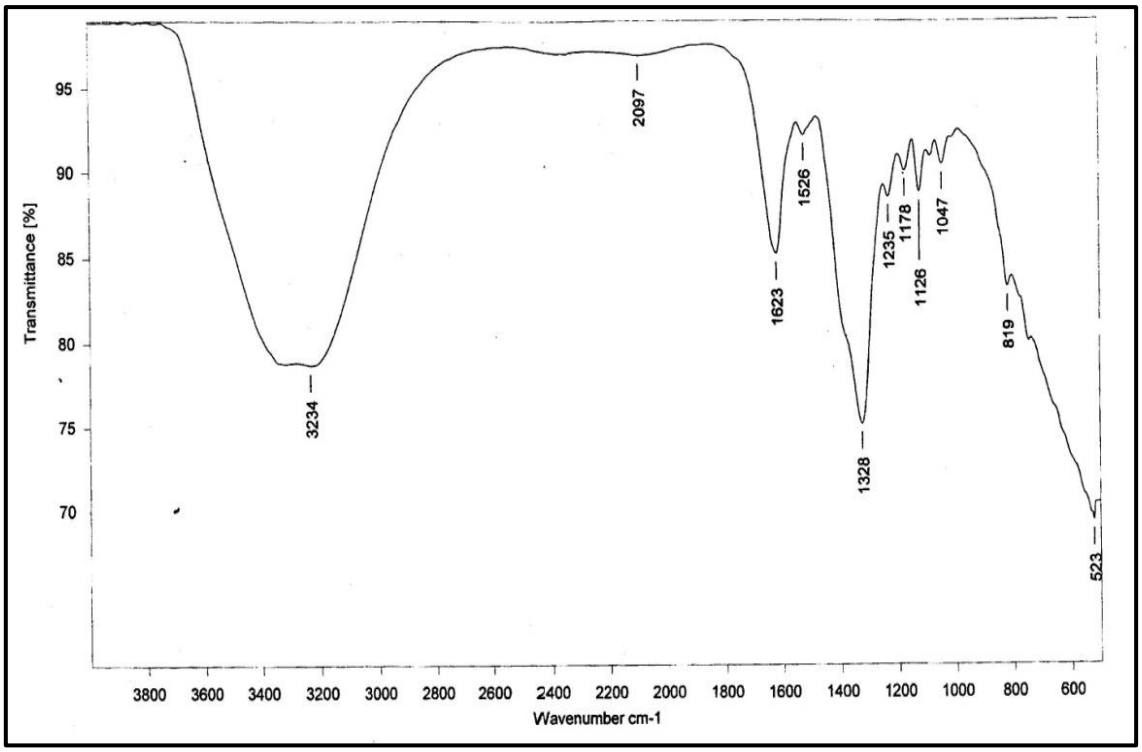


**Figure 2: IR Spectra of Salicyl Sulphaguanidine Nickel (II) complexes[SDN]**

**Table 1. IR Spectra of Salicyl Sulphaguanidine metal (II) complex**

| Compound | ν [C=N] $cm^{-1}$ | ν [O-H] $cm^{-1}$ | ν[SO2]as $cm^{-1}$ | ν [SO2]s $cm^{-1}$ | ν [C=O] $cm^{-1}$ | ν [M-O] $cm^{-1}$ | ν [M-N] $cm^{-1}$ |
|---|---|---|---|---|---|---|---|
| Schiff base | 1618 | 3145 | 1321 | 1159 | 1578 | -- | -- |
| SGC | 1631 | 3232 | 1321 | 1159 | 1524 | 555 | 500 |
| SGCO | 1620 | 3456 | 1322 | 1152 | 1529 | 540 | 522 |
| SGN | 1623 | 3234 | 1328 | 1126 | 1528 | 523 | 515 |

### 3.2 $^{1}$HNMR SPECTRA

Nuclear magnetic resonance spectroscopy is known as NMR spectroscopy. It is a research technique that exploits the magnetic properties of certain atomic nuclei. This spectroscopy determines the physical and chemical properties of atoms and molecules. It explains the phenomenon of nuclear magnetic resonance. It can provide information about the structure, dynamics, reaction state, and chemical environment of

molecules. The intermolecular magnetic field which around an atom in a molecule changes the resonance frequency. It gives the details of the electronic structure of a molecule and its functional groups.

### 3.2.1SALICYL SULPHA GUANIDINE[SG] AND ITS COMPLEXES

In the $H^1$NMR Spectrum of the schiffbase, ligand complexes[SGN] the Azomethine signals were formed 8.733ppm. The azomethine signal in SG Schiff base ligand was formed at 9.02ppm. This azomethine [CH=N] proton was shifted to downfield in the spectra of all the complexes. This is due to the donation of lone pair of electrons by the azomethine nitrogen to the metal atom. The-OH proton signals were formed 12.48-12.50 ppm. This was indicating the involvement of phenolic oxygen coordination in complexes. The $^1$HNMR spectra of ligands in DMSO-$d_6$ revealed a multiplet at 6.40-6.750 ppm corresponding to aromatic protons. Thus the NMR results support the IR inferences. The data of ligand and complexes were presented in table 2 and also in figure 3

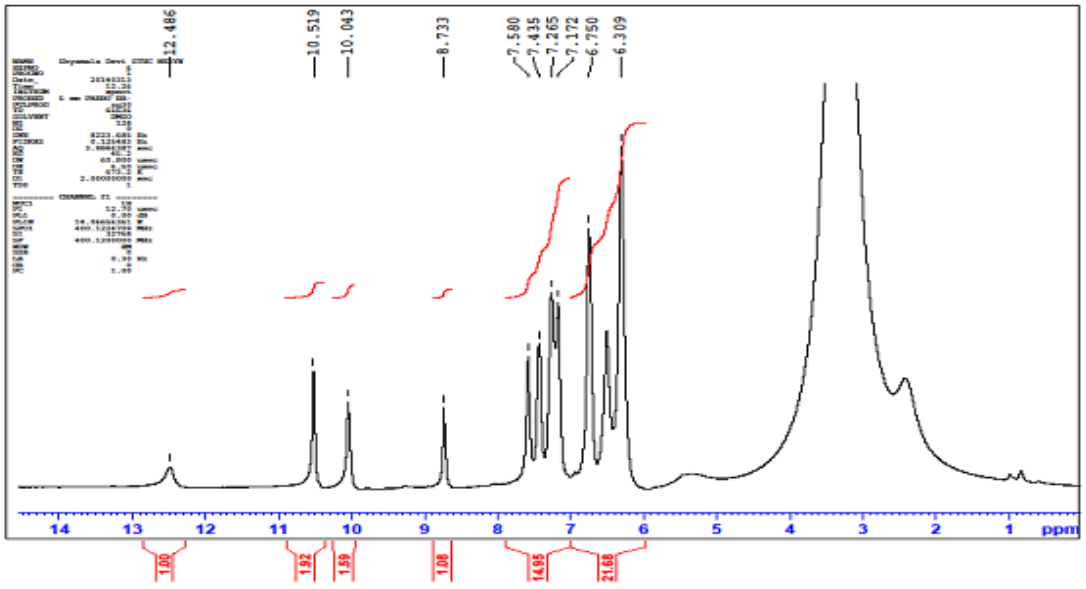


**Figure 3:NMR Spectra of Salicyl Sulphaguanidine Nickel (II) complex[SGN]**

**Table 2 - NMR Spectra of Salicyl Sulphaguanidine (II) complex**

| Complexes | Phenolic [OH] (ppm) | Azomethine (ppm) | Salicyl phenyl (ppm) | $NH_2$ (ppm) | NH (ppm) |
|---|---|---|---|---|---|
| SG | 12.50 | 9.04 | 7.650 | 6.56 | 11.807 |
| SGN | 12.486 | 8.733 | 7.580 | 6.750 | 10.519 |

### 3.3 SALICYL SULPHA GUANIDINE[SG] AND ITS COMPLEXES

The absorbance and their wavelength are given in table 3. and elemental analytical datas were indicated in the table 4. The electronic spectra of the complexes are given in figure 4. The absorption spectrum of Schiff base SG is maximum at 325nm [30769cm$^{-1}$]. On complexation this band was shifted towards lower wavelength region. The nickel complexes exhibit absorption band in the region 250nm [40000cm$^{-1}$]-246nm [40650cm$^{-1}$] is attributed to transition $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ indicating octahedral geometry.

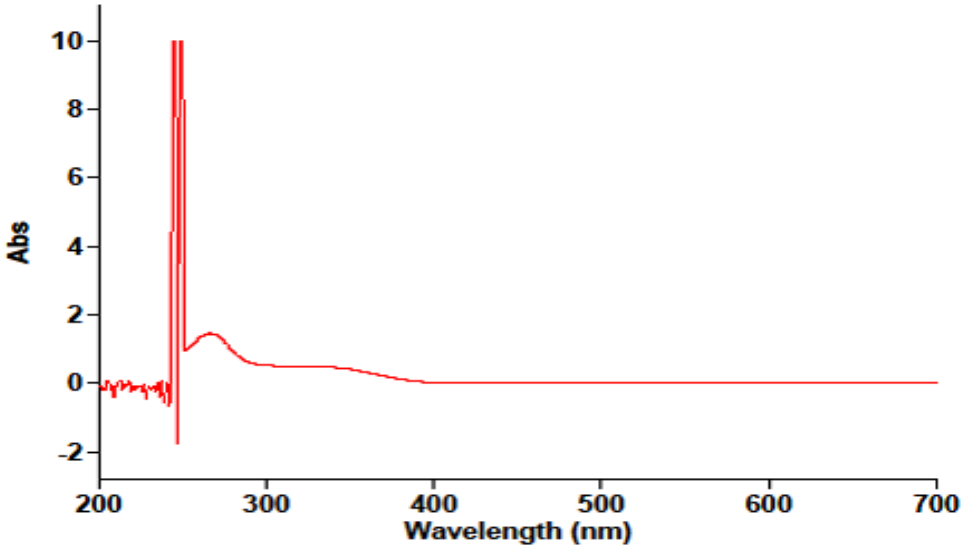


**Figure 4: UV Electronic spectra of Salicyl Sulphaguanidine Nickel (II) complex[SGN]**

**Table 3:UV Electronic spectral data of metal complexes of salicylaldehyde and sulpha guanidine Schiff base**

| Complex | Absorption maximum | | Assignments |
|---|---|---|---|
| | $\lambda_{max}$(nm) | V($cm^{-1}$) | |
| SGN | 250 | 40000 | Intra ligand transition |
| | 266 | 37593 | Intra ligand transition |
| | 246 | 40650 | Charge transfer transition |
| | 325 | 30769 | Charge transfer transition |

**Table 4: Elemental analysis and molar conductivity data of salicyl sulphaguanidine Schiff base complexes**

| S.No | Complexes | Color | Elemental Analysis % [found/ calculated] | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | C | H | N | O | S | M |
| 1 | SG $[C_{14}H_{14}N_4O_3S]$ (336.3) | Yellow | 52.8 49.95 | 4.40 4.16 | 17.6 16.65 | 15.09 14.27 | 10.06 9.51 | -- |
| 2 | SGN $[C_{15}H_{25}N_5O_9SNi]$ (520.1) | Pale yellow | 48.60 (39.91) | 6.32 (5.19) | 17.72 (14.55) | 36.45 (29.93) | 8.70 (6.65) | 7.5 (6.23) |

X-ray powder diffraction study of the synthesized complexes brought about evidence about the structure and atomic spacing of the complexes. The Xray diffraction of Ni[II complexes of salicyl sulphaguanidine Schiff base was scanned in the range of 20-$80^0$ using Cu target as a source of Xrays having wavelength1.54A. The diffractogram and associated data depicted the 2θ value for each peak, relative

intensity and interplanar spacing(d-values). The diffractogram of Ni(II) complex showed reflections with maxima at 2θ:11.11$^0$. Its intensity is 100a.u corresponding to d value 3.1646A$^0$.

The crystalline size of the complexes could be estimated from XRD pattern and it was found to be 0.0502x10$^{-6}$ for Ni(II) and Co(II) . From this it was suggested that the complexes were microcrystalline in nature. X-ray powdered diffraction data of complexe are presented in table 5. The XRD pattern of the complexes are shown in figure 5.

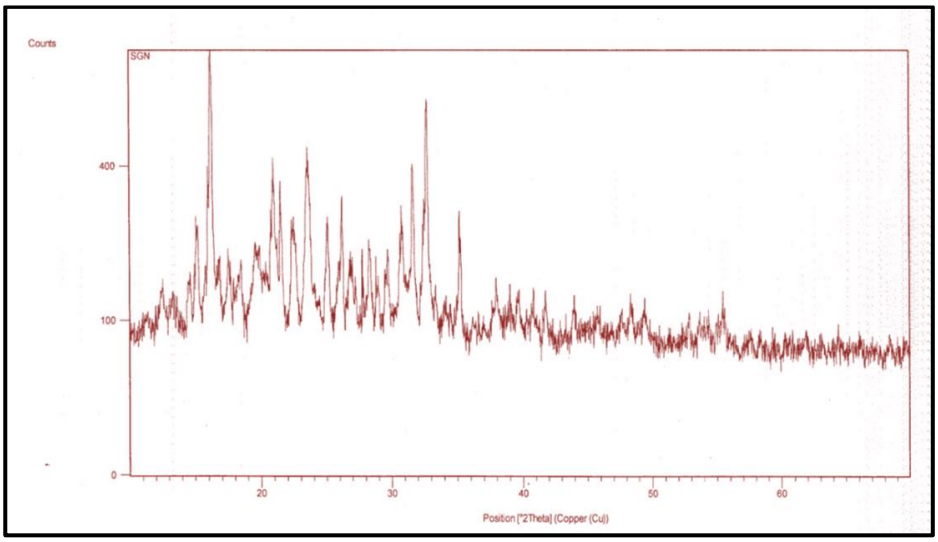


**Figure 5:XRD Spectra of Salicyl Sulphaguanidine Nickel (II) complex**

**Table 5:XRD value of Salicyl Sulphaguanidine Nickel (II) complex**

| Sl.no | 2θ (degree) | θ (degree) | FWHM(β) in radian | D x10$^{-6}$ A$^0$ |
|---|---|---|---|---|
| 1 | 11.36 | 5.68 | 0.0058 | 0.0248 |
| 2 | 12.46 | 6.23 | 0.0046 | 0.0312 |
| 3 | 13.59 | 6.79 | 0.0017 | 0.0834 |
| 4 | 14.42 | 7.21 | 0.0023 | 0.0616 |
| | | | MEAN | 0.0502x10$^{-6}$ |

## 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.

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