

THE INTERNATIONAL JOURNAL OF SCIENCE & TECHNOLEDGE

Synthesis, Spectral Characterization and Antimicrobial Activity of Schiff Base Complexes of Cu [LI] and Co [LI]

M. Shyamala Devi Thankachi

Research Scholar, S.T. Hindu College, Nagercoil, Tamil Nadu, India

S. Suthakumari

Assistant Professor, S.T Hindu College, Nagercoil, Tamil Nadu, India

Abstract:

The complexes [SDC, SDCO] of Cu [II], and Co[II] ions with a Schiff base[SD] derived from salicylaldehyde and sulfadiazine have been investigated. The resulting complexes were characterized by UV/Vis IR, ¹HNMR spectral studies, elemental analysis, molar conductance, XRD, Magnetic susceptibility and antimicrobial activity. The molar conductance measurements indicates that the complexes were non electrolytes. The IR data shows that complexes were mono basic ligand with NO bidentate sites The XRD studies show 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.

Keywords: Schiff base, Sulfadiazine, Antifungal and Antibacterial activities.

1. Introduction

Schiff base compounds which contain the azomethine group (-RC=N-). They are prepared by the condensation of a primary amine with an active carbonyl compound. Schiff bases were otherwise known as antiviral agents [1]. They have a variety of biological activities like antifungal, antibacterial, antimalarial and antipyretic properties. Sulfanomides were used as antitumor [2], diuretic [3], anti thyroid [4] and antibacterial activities [5-7]. The azomethine and sulfanamide group was responsible for antimicrobial activity. They inhibit the growth of bacteria but they do not kill them. Tetradentate Schiff bases were the subject of many authors. We report here for preparation and characterization of some bidentate such as SDC and SDCO. The infra red spectra, NMR spectra, UV spectra, XRD analysis and biological activities of the complex were also carried out.

2. Experimental Section

2.1. Materials and Methods

Salicylaldehyde [Fluka]. Sulfadiazine [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, UV/Vis spectra 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. 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.

2.2. Synthesis of Schiff Base

Added Salicylaldehyde[0.06ml,0.1mmol] in 10ml of absolute ethanol dropwise stirring to sulfadiazine[0.105mg,0.1mmol] in 10ml of ethanol in a beaker with the molar ratio 1:1. The reaction mixture was heated for 24 hours during which the colour of the solution change to yellow. The yellow solid product was filtered and recrystallized in ethanol after 24hours. The melting point was 260°C.

2.3. 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 sulfadiazine. The reaction mixture was heated for 22 hours. The complex precipitate was separated by filtration. Washed with ethanol and dried.

3. Results and Discussion

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

3.1. IR Spectra

The IR spectral data of the ligand showed a band at 1618cm^{-1} which was assigned to ν [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 $511-519\text{cm}^{-1}$ and $450-497\text{cm}^{-1}$ are attributed to [M-O] and M-N] stretching vibrations respectively. The broad band in the region $3145-3355\text{cm}^{-1}$ was accorded to ν [O-H] stretching vibrations a feature indicating the presence of water $1524-1580\text{cm}^{-1}$. The bands $1321, 1159\text{cm}^{-1}$ was not changed because sulfonamide O2 were not contributing in coordination with the metal ions.

Compound	ν [C=N] cm^{-1}	ν [O-H] cm^{-1}	ν [SO ₂]as cm^{-1}	ν [SO ₂]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	511	450
SDC	1631	3232	1321	1159	1524	514	489
SDCO	1649	3355	1321	1152	1580	519	497

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

SDC – Copper complex, SDCO – Cobalt Complex, SD – Schiff base

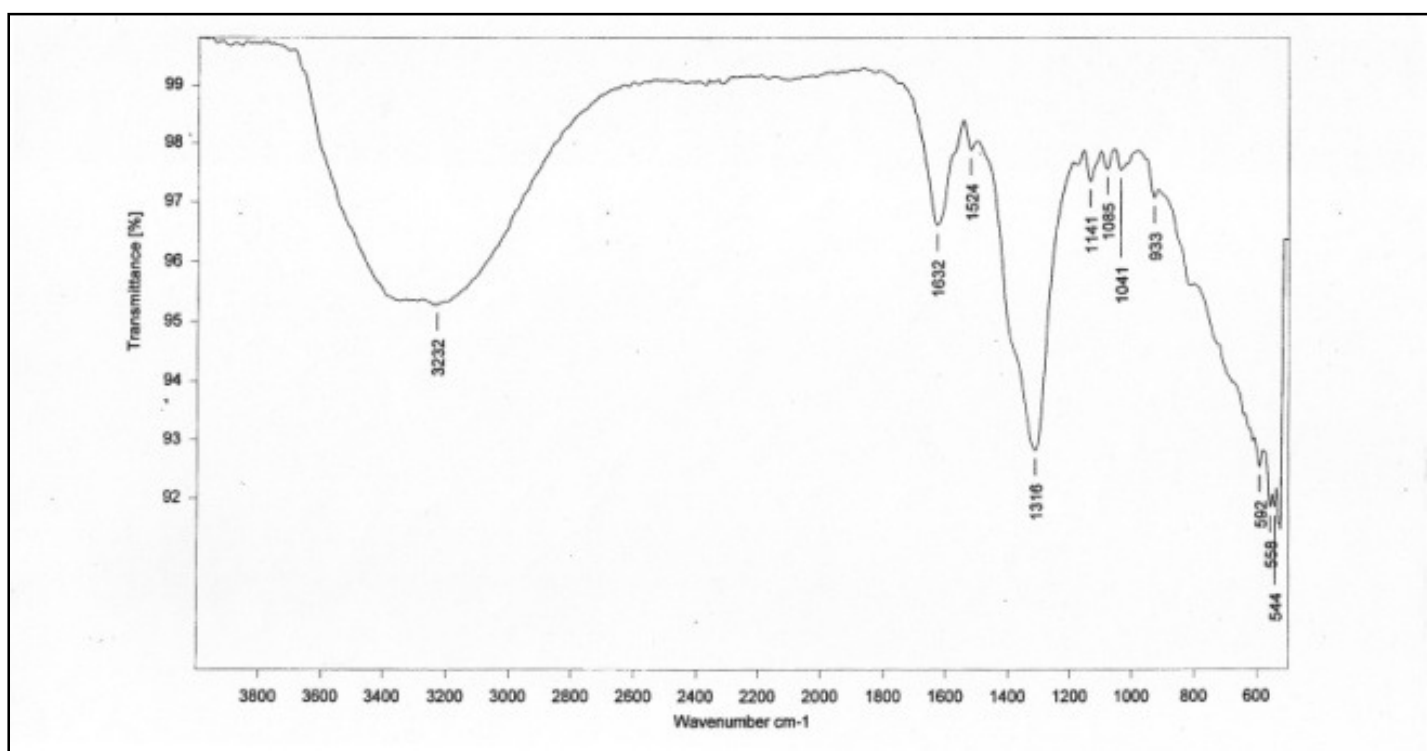


Figure 1: IR Spectra of SDC

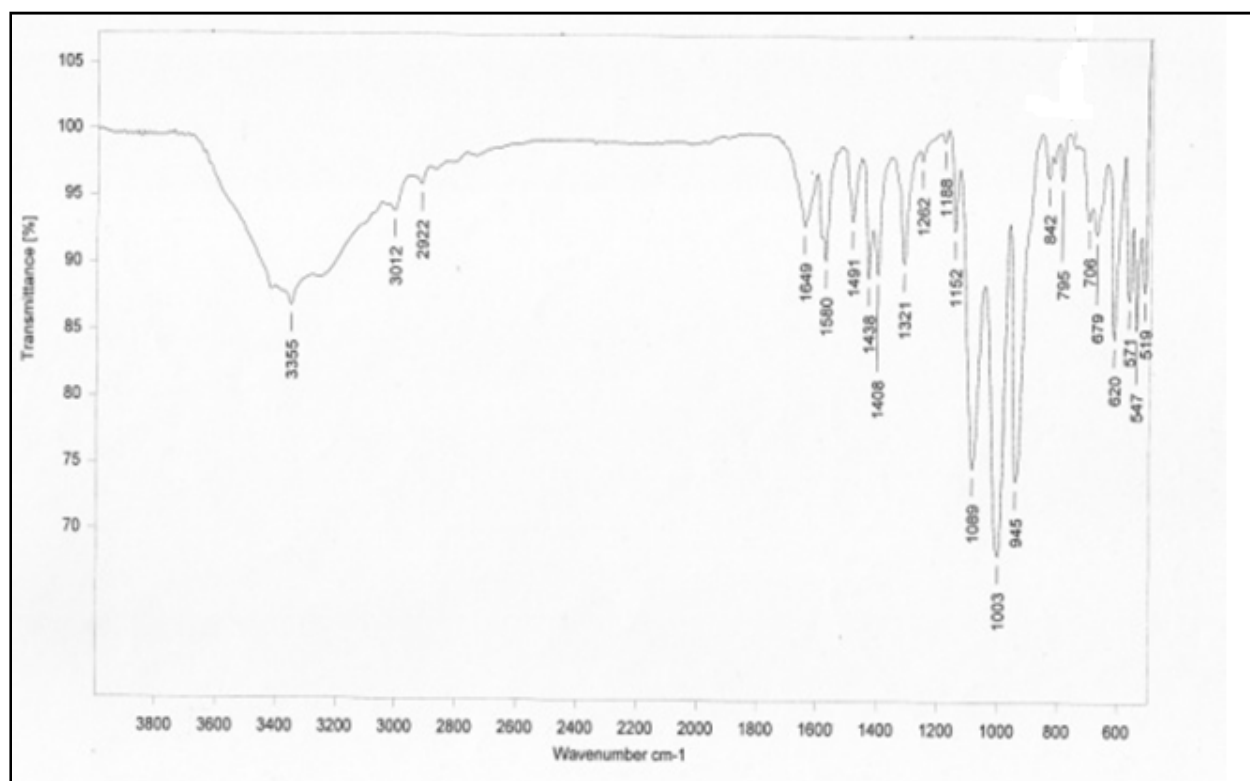


Figure 2: IR Spectra of SDCO

3.2. ¹HNMR Spectra

In the ¹HNMR Spectrum of the Schiff base, The Azomethine signals at 9.00ppm-9.34 in SD, SDC and SDCO. 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 at 12.50ppm. This band was not present in SDCO and SDN. This was indicating the involvement of phenolic O₂ in the coordination's in complexes. The ¹HNMR spectra of ligands in DMSO-d₆ revealed a multiplet at 6.40-6.57ppm corresponding to aromatic protons. Thus the NMR results support the IR inferences.

Compound	Phenolic[OH]	Azomethine	Saly phenyl	NH ₂	NH
SD	12.50	9.04	7.650	6.56	11.807
SDC	12.50	9.04	7.865	6.547	10.757
SDCO	-	9.34	7.610	6.40	10.778

Table 2: The ¹HNMR spectra of Schiff base and complexes

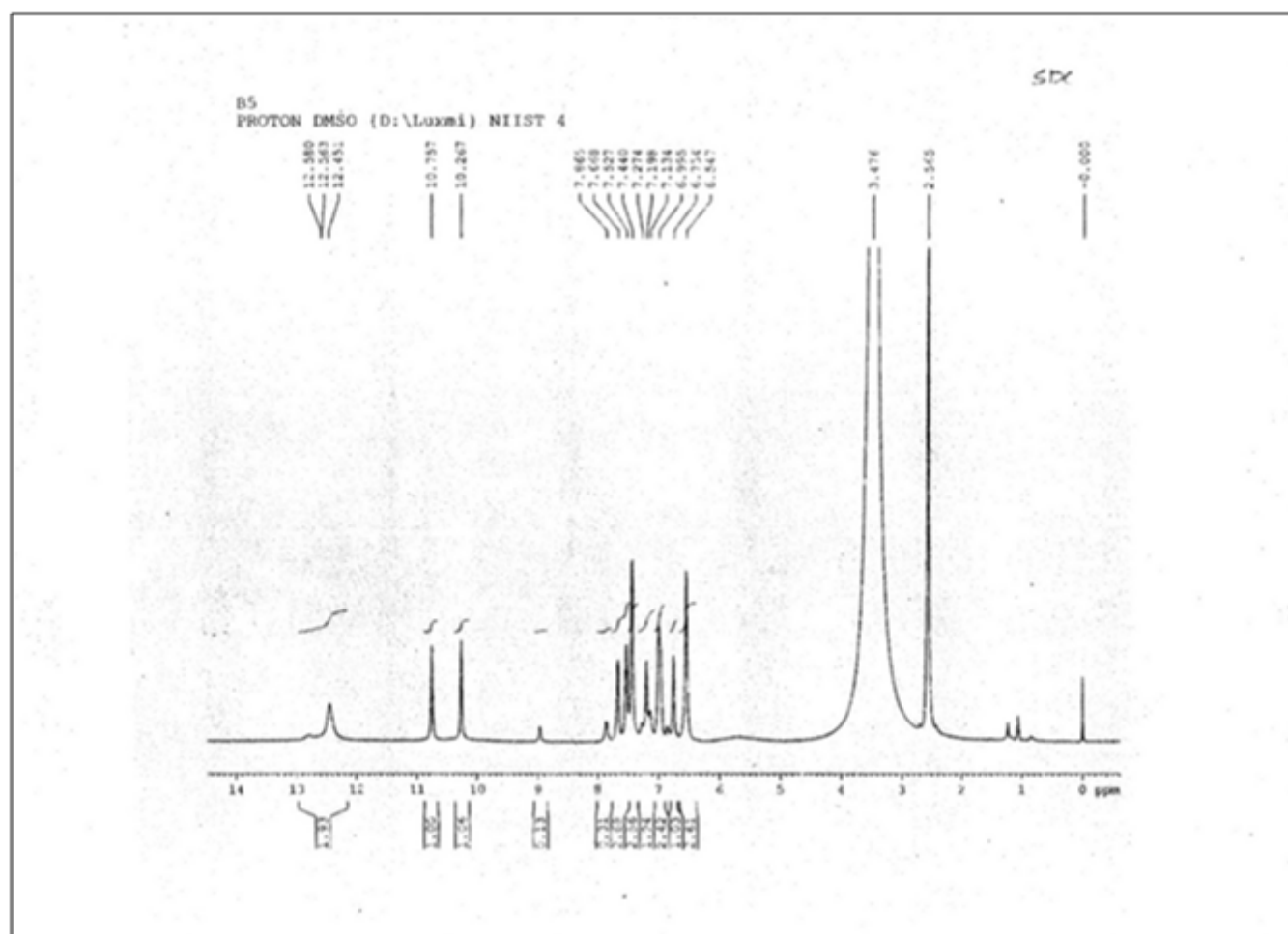


Figure 3: HNMR spectra of SDC

3.3. Elemental Analysis

S. No	Complexes	Color	Magnetic moment	Elemental Analysis % [found/ calculated]			
				C	H	N	S
1	SD[C ₁₅ H ₁₅ N ₄ O ₃ S] (372.378)	Yellow		53.48 (50.87)	3.6 (3.4)	11.69 (11.12)	10.06 (9.51)
2	SDC[C ₁₉ H ₂₀ N ₄ O ₉ SCu] (435.918)	Dark green	1.72	54.96 (49.00)	5.85 (5.21)	10.68 (9.52)	16.28 (14.51)
3	SDCO[C ₁₉ H ₂₆ N ₄ O ₉ SCo] (405.34)	Black	4.24	51.18 (49.50)	5.4 (5.27)	9.95 (9.62)	11.37 (11.00)

Table 3: Analytical Data of the ligand and its complexes

3.4. Conductance Measurements

The observed molar conductance was 6.9-12.0 $\text{Ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ 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 [8].

3.5. UV SPECTRA

The solution electronic spectra of the ligand and the complexes were recorded DMSO solvent in the u.v visible region an absorption band found at 250nm-256nm. The electronic spectra of the metal complexes recorded in Region exhibit intraligand and charge transfer transition of the C=N chromophore. On complexation this band was shifted to wavelength region at 294nm and 275nm suggesting the coordination of azomethine nitrogen with Cu[II] and Co[II] ions. ligands. The spectra also show the other transitions in the range of 340-400nm, which can be assigned to $n-\pi^*$ 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 [9-10].

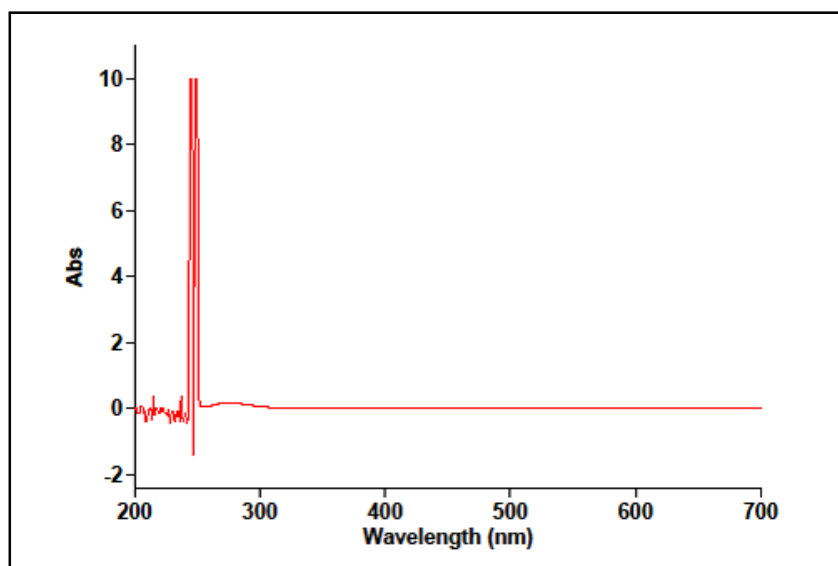


Figure 4: UV Spectra of Salicylaldehyde and Sulphadiazine copper (II) complex

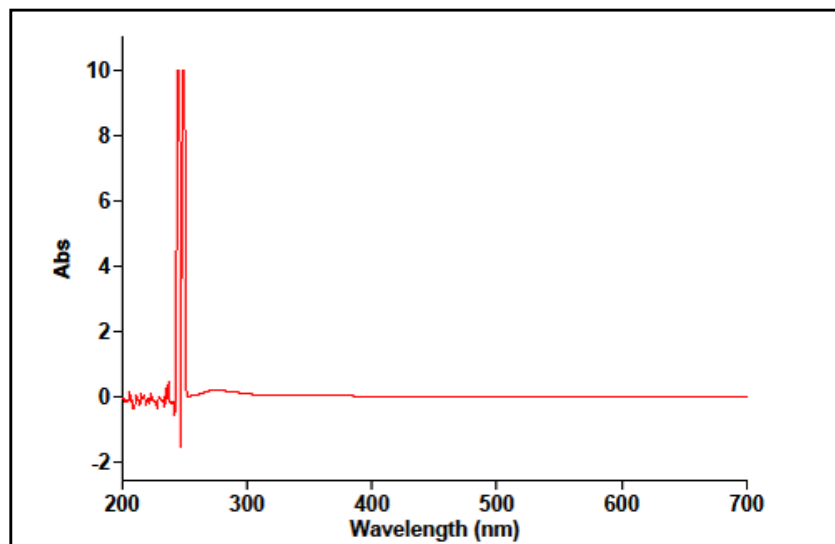


Figure 5: UV Spectra of Salicylaldehyde and Sulphadiazine cobalt (II) complex

3.6. XRD

The crystalline structure was performed by X ray diffraction analysis[XRD] utilizing Cu-K α [wavelength1.5406 \AA] radiation Using the Scherer formula the crystallite size of the prepared complexes of SDC, SDCO have been calculated. Scherer formula can be written as follows.

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

λ is the wavelength of x ray used

β is the full width at half maximum

θ is the diffraction angle

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

2 θ	Θ	FWHM(β) in radian	D $\times 10^{-6}$
28.26	14.13	0.0017	0.0802
29.34	14.67	0.0035	0.0400
		MEAN	0.0601 $\times 10^{-6}$

Table 4: XRD value of Salicylaldehyde and Sulphadiazine copper (II) complex

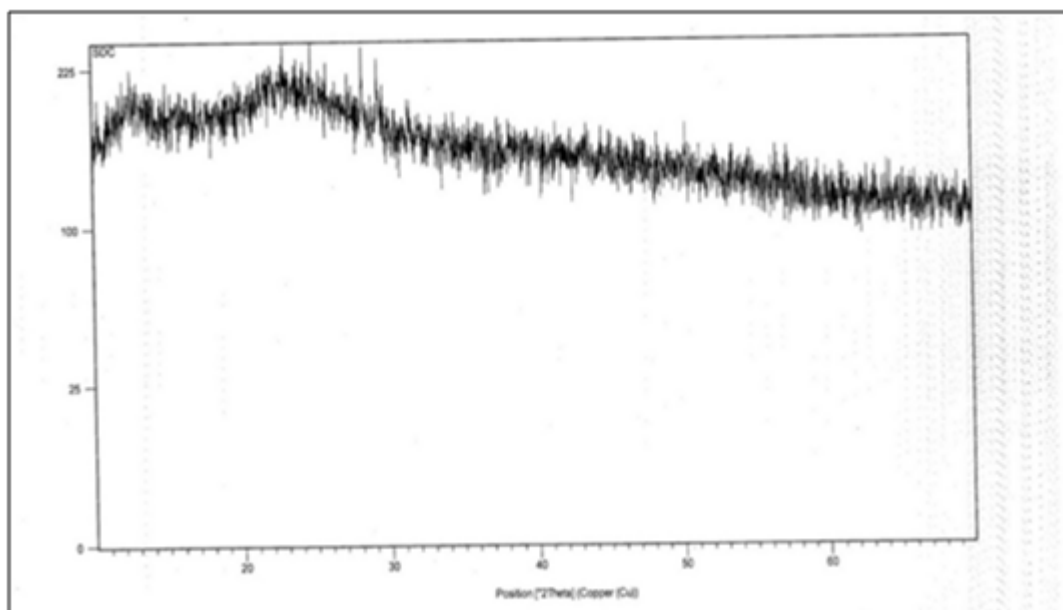


Figure 6: XRD Spectra of Salicylaldehyde and Sulphadiazine Copper (II) complex

2θ	θ	FWHM(β) in radian	D x10 ⁻⁶
31.37	15.68	0.0042	0.0351
21.38	10.69	0.0037	0.0398
22.67	11.33	0.0036	0.0394
28.23	14.11	0.0048	0.0292
MEAN			0.0358 x10 ⁻⁶

Table 5: XRD Spectra of Salicylaldehyde and Sulphadiazine cobalt (II) complex

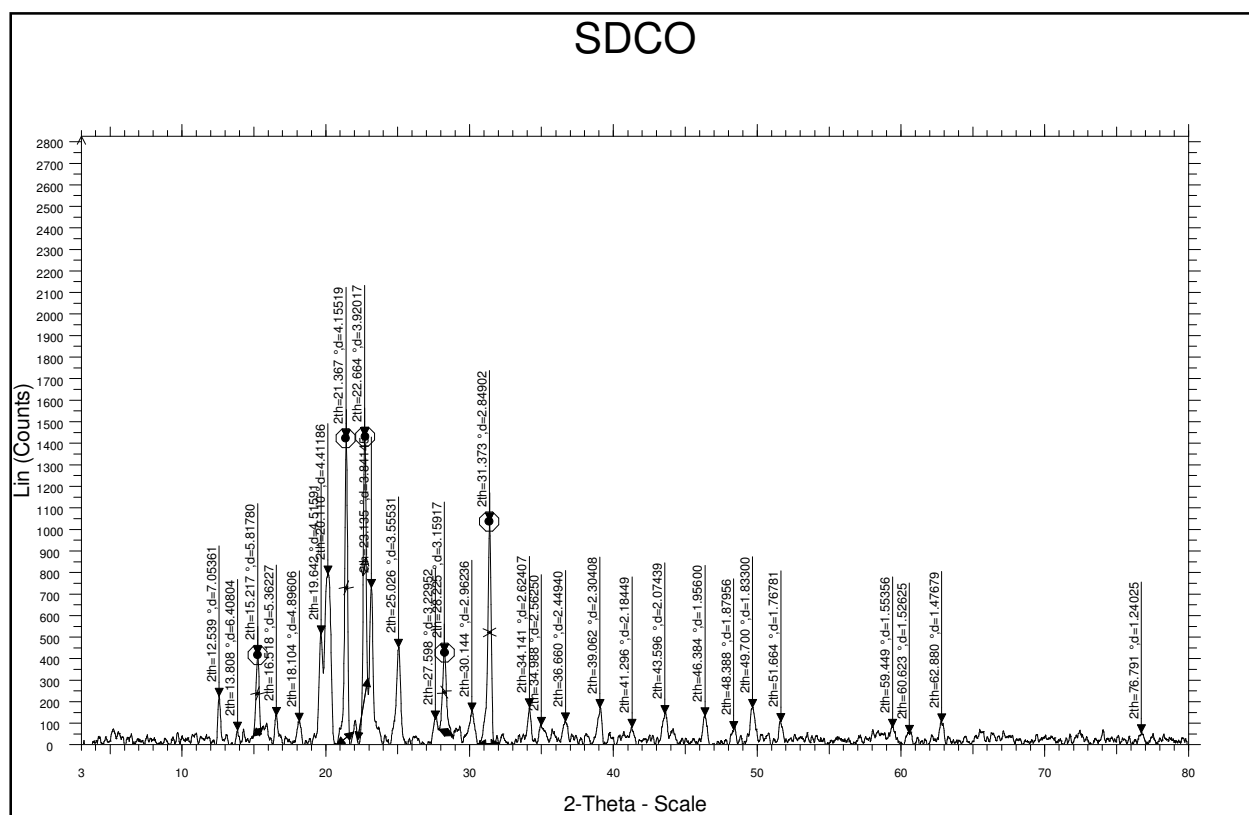


Figure 7: XRD Spectra of Salicylaldehyde and Sulphadiazine cobalt (II) complex

3.7. Biological activity

Antifungal and anti bacterial activities of the sulfadiazine 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°C. The results were indicated that in the table 8.

S. no	Organisms	Media	Chloram phenicol	SD	SDC	SDCO	NC
1	E. coli	Muller Hinton Agar	23.0	6.0	14.0	19.0	6.0
2	Pseudomonas aeruginosa		21.0	6.0	17.0	6.0	6.0
3	S. aureus		22.0	6.0	10.0	6.0	6.0
4	Candida		22.0	6.0	14.0	6.0	6.0

Table 6: Zone of Inhibition (mm)

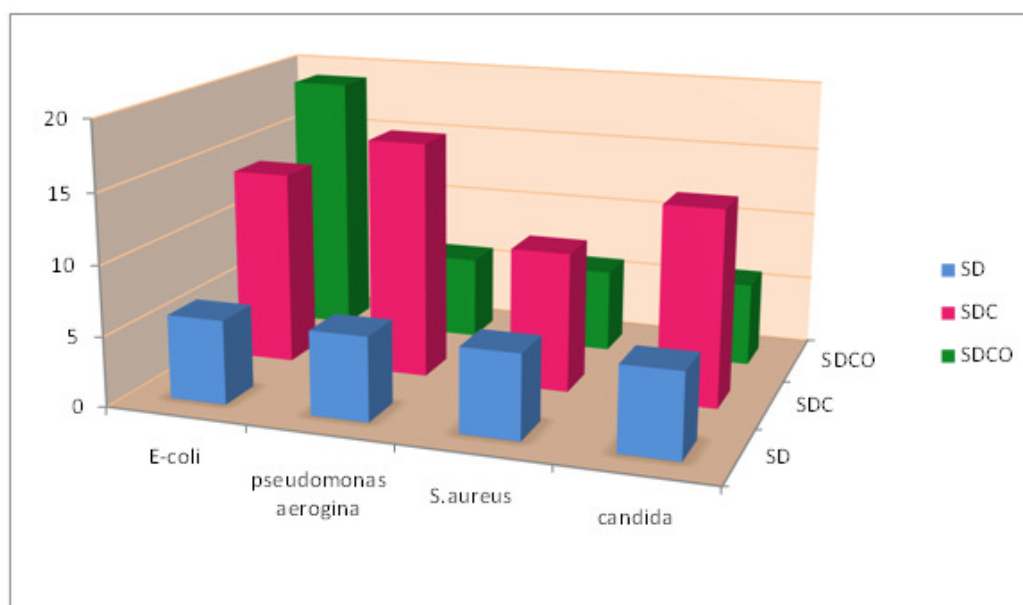


Figure 8: Antimicrobial activity of Schiff base

4. 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 SDC complex was least active and the complex of SDCO was highly active in E. coli. This effect may be due to the co ordination of metal ion. They killed the cells of bacteria and fungi so they can be used as anti bacterial and antifungal agents.

5. Acknowledgement

Authors owe the deepest gratitude to The Management, Principal and HOD of chemistry S.T. Hindu College, Nagercoil who made us available the support in a number of ways for completing this research project successfully.

6. References

- i. Cheng L, Tang J, Luo H, Jin X, Dai F, Yang J, Qian Y, Li X, Zhou (2010). B: Antioxidant and antiproliferative activities of hydroxyl-substituted Schiff bases. Bioorg. Med. Chem. Lett, 20, 2417-2420.
- ii. Domagk G (1935). Chemotherapy of bacterial infections. Deut Med Wochensch, 61.
- iii. Boyd A (1988). Sulfonamide receptors ion channels and fruit flies. Diabetes, 37, 847-850.
- iv. Ogden RC, Flexner C (2001). Protease inhibitors in AIDS therapy.
- v. Mandell GL, Peri WA, Hardman JG, Limbid LE, Molinoff PB, Ruddon RW, Gilman AG (1966). Pharmaceutical basis of therapeutics, 9th edition, New York McGraw_Hill.
- vi. Maren TH (1976) Relations between structure and biological activity of sulfonamides. Annu Rev Pharmacol Toxicol, 16,309-327.
- vii. Owa T, Nagasu T (2001). Novel Sulfonamide derivatives for the treatment of cancer. Exp Opin Ther Pat, 10, 1725-1740.
- viii. W J Geary (1971). Coord Chem Rev, 7.
- ix. Lever, A.B.P(1984) Inorganic Electronic spectroscopy, Elsevier.Amsterdam.
- x. Chakravarthi P.B., Khanna P.J(1985). Ind.Chem.soc.