



**SYNTHESIS, SPECTRAL AND THERMAL STUDIES OF SCHIFF BASE
COMPLEXES DERIVED FROM N-(4-((2-HYDROXYBENZYLIDENE) AMINO)
PHENYL) PYRIDINE-2-SULFONAMIDE**

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ABSTRACT:

Coordination complexes of Co(II), Ni(II) and Cu(II) have been synthesized from N-(4-((2-hydroxybenzylidene) amino) phenyl) pyridine-2-sulfonamideschiff base derived from sulphapyridine and salicylaldehyde. The nature of bonding and structural features of the Schiff base and its complexes have been deduced from elemental analysis, molar conductance, magnetic susceptibility measurements, spectral studies (IR, ¹H NMR, UV-Visible) mass, TGA. The spectral data of the complexes have revealed bidentate coordinating nature of the Schiff base through azomethine nitrogen atom and phenolic oxygen. Elemental analysis data suggest 1:1 [M: L] stoichiometry ratio in all metal complexes. The molar conductance measurements reveal the non-electrolytic nature of complexes. The electronic spectral data support square planar geometry for all synthesized complexes. The thermal degradation patterns of the compounds have been analyzed from the thermograms and the kinetic parameters have been evaluated.

KEYWORDS- sulphapyridine, salicylaldehyde, metal complexes, Schiff base

INTRODUCTION:

Numerous investigations have been conducted in the past on various Schiff base metal complexes due to their extensive range of applicationsⁱ. Schiff bases and their metal complexes play a crucial role in our comprehension of the coordination chemistry of transition metal ions^{ii-iv}. There is significant interest surrounding the chemistry of transition metal complexes that consist of ligands containing oxygen, nitrogen and sulfur donor atoms, owing to their antitumor, antiviral, antifungal and antibacterial properties^{iv}. Considerable interest has been sparked in the coordination chemistry of metal complexes derived from Schiff bases of sulfa drugs due to their prominent biological activity. These biologically active compounds, formed through the condensation of sulfa drugs with aldehydes and ketones, also have ability to form complexes with metal ions^{iv}. There has been a surge in research focused on sulfonamides and their transition metal complexes due to their promising applications in the field of chemistry, biology and medicine^{vii-ix}.

The literature has not extensively explored the synthesis of compounds that incorporate both Schiff base and sulfonamide fragments. Numerous Schiff bases derived from sulfa drugs have, however, been synthesized and employed as ligands for the preparation of highly effective metal complexes^{x-xii}.

EXPERIMENTAL:

MATERIALS AND METHODS

All the solvents used were of analytical reagent grade purchased from SD Fine and Merck. Pure sample of dapsone was obtained from Shah Scientific Pvt. Ltd. Mumbai. Solvents were purified and dried before use by literature method^{xiii}. The ligand used in the present work is not commercially available; hence, it was synthesized in the laboratory.

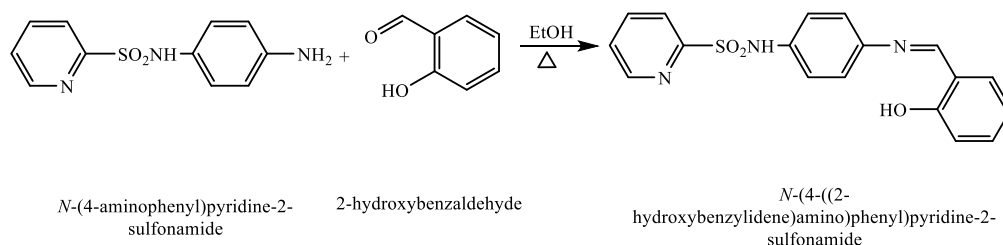
Microanalyses of carbon, hydrogen and nitrogen of the ligands were carried out on a Perkin Elmer CHN 2400 elemental analyzer. ¹H-NMR spectra of the ligands were recorded in DMSO-d₆ solution on EM-360, 60 MHz NMR Spectrometer. The electronic spectra of the ligands were recorded on a Shimadzu UV/Vis spectrophotometer in the region 200-1000 nm. The Fourier-transform infrared (FTIR) spectra of ligands were recorded as KBr pellets using Shimadzu spectrometer (IRAffinity-1S). Mass spectra of synthesized compounds were carried out on Thermo scientific TSQ 8000 Gas Chromatograph- Mass Spectrometer.

GENERAL PROCEDURE

FOR THE PREPARATION OF N-(4-((2-HYDROXYBENZYLIDENE) AMINO) PHENYL) PYRIDINE-2-SULFONAMIDE(SCHEME-1)

Equimolar (0.01m) solutions of 2-hydroxybenzaldehyde (2ml) and N-(4-aminophenyl) pyridine-2-sulfonamide (2.49g) were mixed separately in ethanol (50 ml) and refluxed for 3 hours. The solution was concentrated and cooled. Orange colored precipitate was obtained which was filtered off and recrystallized from ethanol. The purity of the compounds were checked by thin layer chromatography (TLC).

Yield- 81 %, M. P. 148⁰C



(Scheme-1)

PREPARATION OF METAL COMPLEXES

The complexes are generally synthesized by refluxing respective metal salt with ligand in ethanolic medium for desired time. The metal salts used for the preparation of complexes were cobalt(II) acetate tetrahydrate [Co(OAc)₂.4H₂O], Nickel(II) acetate tetrahydrate [Ni(OAc)₂.4H₂O] and Copper(II) acetate monohydrate [Cu(OAc)₂.H₂O].

FOR THE PREPARATION OF Co(II) COMPLEXES

Cobalt(II) acetate tetrahydrate [Co(OAc)₂.4H₂O] (0.02 mol, 4.75g) and ligands (0.02mol) were dissolved separately in absolute alcohol. Both the solutions were filtered and mixed in hot conditions. Few drops of 1N KOH were added to the reaction mixture and then the reaction was refluxed for about 8-10 hours. The colored solid obtained were filtered off, washed several times with small amount of ethanol followed by petroleum ether and finally dried over fused calcium chloride.

FOR THE PREPARATION OF Ni(II) COMPLEXES

Nickel(II) acetate tetrahydrate $[\text{Ni}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}]$ (0.02 mol, 4.97 g) and ligands (0.02 mol) were dissolved separately in absolute alcohol. Both the solutions were filtered and mixed in hot conditions. Few drops of 1N KOH were added to the reaction mixture and then the reaction was refluxed for about 8-10 hours. The colored solid obtained were filtered off, washed several times with small amount of ethanol followed by petroleum ether and finally dried over fused calcium chloride.

FOR THE PREPARATION OF Cu(II) COMPLEXES

Copper(II) acetate tetrahydrate $[\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}]$ (0.02 mol, 3.99 g) and ligands (0.02 mol) were dissolved separately in absolute alcohol. Both the solutions were filtered and mixed in hot conditions. Few drops of 1N KOH were added to the reaction mixture and then the reaction was refluxed for about 10-12 hours. The colored solid obtained were filtered off, washed several times with small amount of ethanol followed by petroleum ether and finally dried over fused calcium chloride.

Table 1: Elemental analysis, colour and formula weight of the compound

Compound	Formula wt.	Found (Calc.) %			
		C	H	N	M
$\text{C}_{18}\text{H}_{15}\text{N}_3\text{O}_3\text{S}$	353.08	61.18 (60.63)	4.28 (4.12)	11.89 (11.72)	-
[CoL]	470.37	51.07 (50.21)	3.64 (3.52)	8.93 (8.84)	12.53 (12.49)
[CuL]	474.98	50.57 (50.33)	3.61 (3.49)	8.85 (8.62)	13.38 (13.30)
[NiL]	470.13	51.10 (50.91)	3.64 (3.55)	8.94 (8.72)	12.48 (12.45)

RESULTS AND DISCUSSION

The newly synthesized ligands are characterized by elemental analysis, IR, ^1H NMR, mass and repeated M. P. determination studies. The C, H and N analyses of the ligand satisfactorily coincide with its proposed molecular formula. In order to find out the binding modes (donor sites) towards the metal ion, the Infrared Spectra of the ligands were recorded. The absorption band of donor atom either disappeared or showed increase/ decrease in absorption frequencies due to formation of the complex.

IR Spectra

The IR spectra of metal complexes of LH shows a broad band that appeared in the spectra of the ligand and its metal complexes in the range $3305\text{-}3398\text{ cm}^{-1}$ which is assigned to the stretching vibration of $\nu(\text{N-H})^{\text{xiv}}$. The IR spectral band of the free ligand and its metal complexes indicate that the band at 3235 cm^{-1} due to intramolecular hydrogen bonded $\nu(\text{OH})$ group in the free ligand spectrum disappeared in all complexes suggesting the coordination of ligand through phenolic oxygen via deprotonation^{xv}. Further the bands at 1618 cm^{-1} (azomethine $\text{C}=\text{N}$) shifted its position on complexation^{xvi}.

The bands at 1358 cm^{-1} and 1132 cm^{-1} in the ligand are assigned to $\nu_{\text{as}}(\text{SO}_2)$ and $\nu_{\text{s}}(\text{SO}_2)^{\text{xvii-xix}}$ respectively. These bands remain almost at the same position in all the complexes suggesting that the sulfonyl oxygen is not taking part in coordination. The $\nu(\text{M-N})$ and $\nu(\text{M-O})$ bands have been assigned in the region $505\text{-}543\text{ cm}^{-1}$ and $445\text{-}459\text{ cm}^{-1}$ respectively^{xx}. The coordination of water in Ni(II), Cu(II) and Co(II) complexes is indicated by appearance of

bands at 3414- 3460 cm^{-1} , 1544-1546 cm^{-1} , 820-840 cm^{-1} , 756-771 cm^{-1} assignable to $\nu(\text{OH})$,

Sr. No.	Compound	$\nu(\text{O-H})$ hydrogen bonded	$\nu(\text{NH})$	$\nu(\text{C=N})$	$\nu(\text{SO}_2)$		$\nu(\text{M-O})$	$\nu(\text{M-N})$	$\nu(\text{H}_2\text{O})$
					asym	sym			
1	LH	3235	3398	1618	1358	1132	--	--	--
2	[CoL]	--	3305	1602	1355	1122	543	453	3414,1544, 837, 771
3	[CuL]	--	3357	1589	1332	1125	505	459	3440,1546, 820, 756
4	[NiL]	--	3348	1598	1324	1127	524	445	3460,1543, 840,767

$\delta(\text{OH})$, $\rho(\text{H}_2\text{O})$, $\rho(\text{H}_2\text{O})$ mode respectively^{xxi-xxii}.

Table 2: Infrared spectral data (cm^{-1}) of ligand LH and its complexes

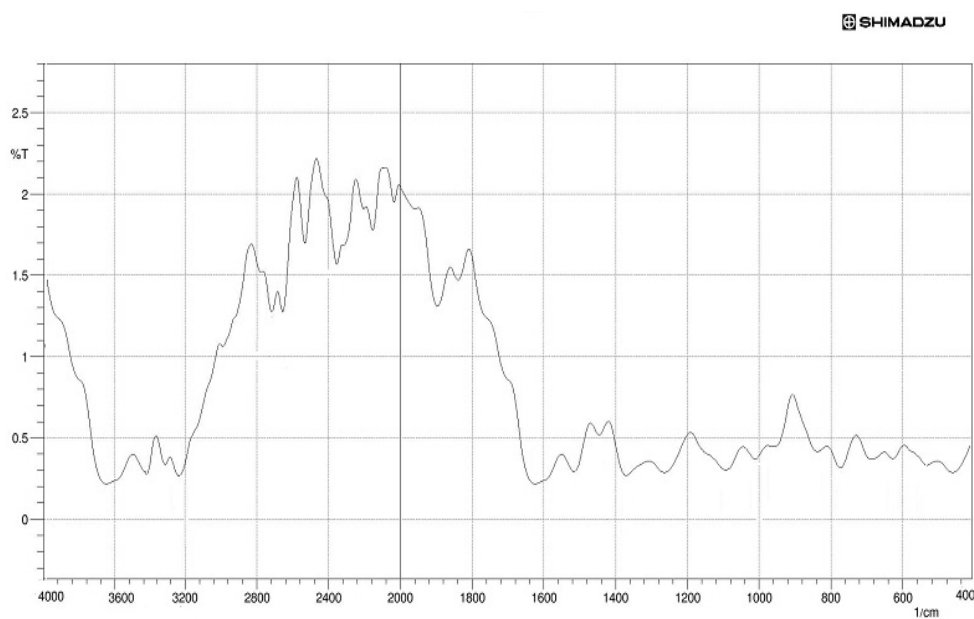


Figure 1: IR spectrum of Co(II) LH complex.

Table 3: $^1\text{H-NMR}$ signals obtained for Ligand LH

Signal position (δppm)	Relative number of protons	Multiplicity	Inference
12.8	1H	Singlet	-OH
8.69	1H	Singlet	Azomethine proton
5.90-7.69	12H	Multiplet	Aromatic proton

Electronic spectra and magnetic moment:

The electronic absorption spectra of metal complexes were recorded in DMSO. UV spectrum of Co(II) complex exhibits absorption bands at 425 nm and 610 nm, which may be assigned to $^2\text{A}_{1g} \rightarrow ^2\text{B}_{1g}$ and $^2\text{A}_{1g} \rightarrow ^2\text{E}_g$ transition respectively. These bands clearly suggest square planar

geometry for Co(II) complex^{xxiii}. Electronic spectra of Cu(II) complex display bands at 440, 554, 675 and 910 nm which are assigned to transition LMCT, ${}^2B_{1g} \rightarrow {}^2A_{1g}$, ${}^2B_{1g} \rightarrow {}^2B_{2g}$ and ${}^2B_{1g} \rightarrow E_g$ respectively. These are consistent with square planar geometry for Cu(II) complex. Electronic spectra of Ni(II) complexes display two bands in the range 622 and 746 nm which are assigned to transition ${}^1A_{1g} \rightarrow {}^1A_{2g}$ and ${}^2A_{1g} \rightarrow {}^2B_{1g}$ transition respectively. All these transitions clearly indicate square planar geometry for Ni(II) complexes^{xxiv}. The magnetic moments, absorption region, band assignment and proposed geometry of the complexes are given in Table 3.

Table 4: Electronic spectral data of the complexes

Complex	Absorption region (nm)	Band assignment	μ_{eff} B. M.	Λ_M $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$	Geometry
[Co(L)]	425	${}^2A_{1g} \rightarrow {}^2B_{1g}$	2.26	7.90	Square planar
	610	${}^2A_{1g} \rightarrow {}^2E_g$			
[Cu(L)]	440	LMCT	0.80	8.58	Square planar
	554	${}^2B_{1g} \rightarrow {}^2A_{1g}$			
	675	${}^2B_{1g} \rightarrow {}^2B_{2g}$			
	910	${}^2B_{1g} \rightarrow E_g$			
[Ni(L)]	622	${}^1A_{1g} \rightarrow {}^1A_{2g}$	Dia.	7.25	Square planar
	746	${}^2A_{1g} \rightarrow {}^2B_{1g}$			

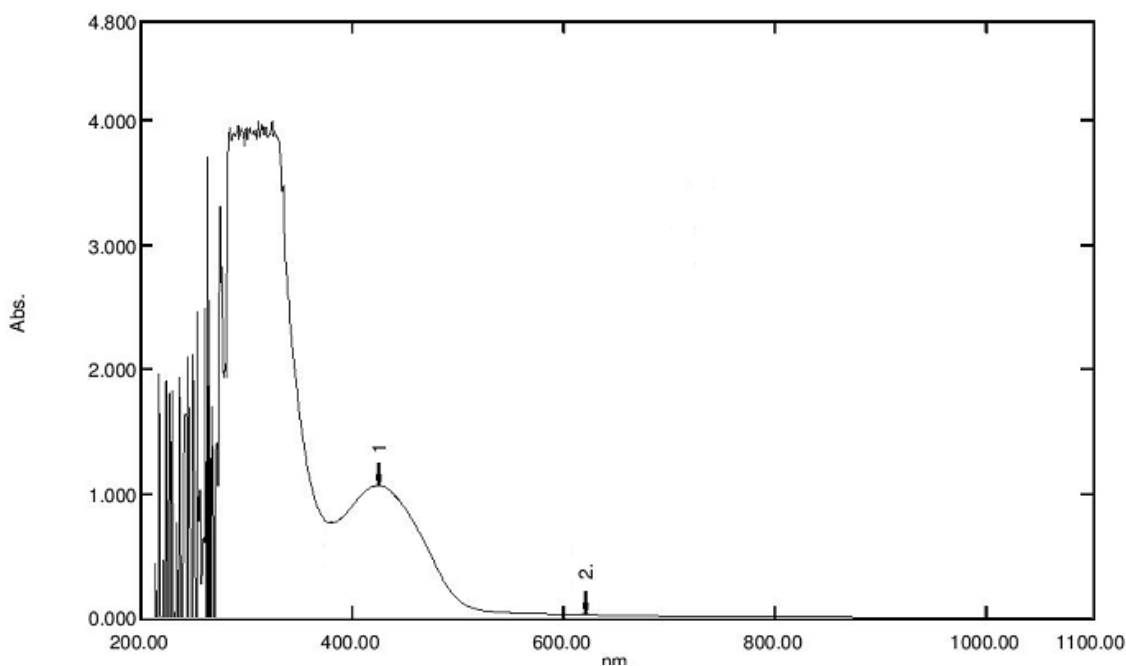


Figure 2: UV-Vis spectra of Co(II) LH complex.

Thermal analysis

An analysis of TG curves of LH and its complexes reveals a four stage decomposition pattern for Co(II), Ni(II), Cu(II) complexes. The TG curve of Co(II), Ni(II) and Cu(II) complexes are stable upto 60°C. Decomposition of one lattice water was observed for Co(II), Ni(II) and Cu(II), complexes at 120°C with % weight loss obs/calcd: Co(II): 3.10/3.05, Ni(II): 3.06/3.01, Cu(II): 3.05/2.99. The further weight loss was observed for Co(II), Ni(II) and Cu(II) from 120-220°C indicates the presence of one coordinate water molecule in Co(II), Ni(II) and Cu(II) complexes. In all the complexes continuous and rapid weight loss has been observed above 220°C corresponding to thermal degradation of free part of the coordinated

ligand along with the other groups present there in. A gradual weight loss above 340°C corresponds to the degradation of actual coordination part of the ligand. Finally the horizontal level beyond 440°C suggests the formation of final decomposition products corresponding to respective metal CoO, NiO and Cu₂O. The thermal decomposition data of the compounds are given in Table 4. The relative thermal stability on the basis of half decomposition temperature is found to be Co(II) > Ni(II) > Cu(II) > LH.

Table 5: Thermodynamic data of LH and its complexes

Compounds	Half Decomposition Temp.(°C)	Activation Energy Ea (kJ)		Order of Reaction (n)	Entropy Change ΔS (J/mol/K)	Free Energy Change ΔF (kJ/mol)
		FC	SW			
LH	220	24.38	24.44	0.94	-268.22	111.69
[CoL]	450	15.10	14.67	0.93	-222.56	87.86
[CuL]	320	13.77	13.53	0.96	-306.807	110.26
[NiL]	360	16.35	16.31	0.94	-313.24	117.72

Mass spectra of the ligands

Mass spectrometry has been successfully used to determine the molecular ion peak for Schiff base ligand. The various fragmentation peaks obtained for ligand, Co(II), Cu(II) and Ni(II) complexes are in good agreement with proposed structure. Mass spectrum of Co(II) LH complex was shown in Figure 3.

Compound	m/z Value	Fragmented ion
[Co(L)]	599, 581, 486, 426, 331, 272, 248, 213	[C ₂₆ H ₂₅ N ₃ CoO ₈ S] ⁺ , [C ₂₆ H ₂₃ N ₃ CoO ₇ S] ⁺ , [C ₂₄ H ₁₈ N ₃ CoO ₃ S] ⁺ , [C ₁₈ H ₁₄ N ₃ CoO ₄ S] ⁺ , [C ₁₅ H ₁₅ NC ₁₀ O ₄] ⁺ , [C ₁₃ H ₁₀ NC ₁₀ O ₂] ⁺ , [C ₁₁ H ₁₁ N ₃ O ₂ S] ⁺ , [C ₁₃ H ₁₁ NO ₂] ⁺
[Cu(L)]	603, 583, 524, 490, 445, 336, 273, 249	[C ₂₆ H ₂₅ N ₃ CuO ₈ S] ⁺ , [C ₂₆ H ₂₃ N ₃ CuO ₇ S] ⁺ , [C ₂₄ H ₂₀ N ₃ CuO ₅ S] ⁺ , [C ₂₄ H ₁₈ N ₃ CuO ₃ S] ⁺ , [C ₂₄ H ₁₉ N ₃ O ₄ S] ⁺ , [C ₁₅ H ₁₅ NCuO ₄] ⁺ , [C ₁₃ H ₁₀ NCuO ₂] ⁺ , [C ₁₁ H ₁₁ N ₃ O ₂ S] ⁺
[Ni(L)]	598, 579, 520, 445, 425, 332, 271, 249, 215	[C ₂₆ H ₂₅ N ₃ NiO ₈ S] ⁺ , [C ₂₆ H ₂₃ N ₃ NiO ₇ S] ⁺ , [C ₂₄ H ₂₀ N ₃ NiO ₅ S] ⁺ , [C ₂₄ H ₁₉ N ₃ O ₄ S] ⁺ , [C ₁₈ H ₁₄ N ₃ NiO ₄ S] ⁺ , [C ₁₅ H ₁₅ NNiO ₄] ⁺ , [C ₁₃ H ₁₀ NNiO ₂] ⁺ , [C ₁₁ H ₁₁ N ₃ O ₂ S] ⁺ , [C ₁₃ H ₁₁ NO ₂] ⁺

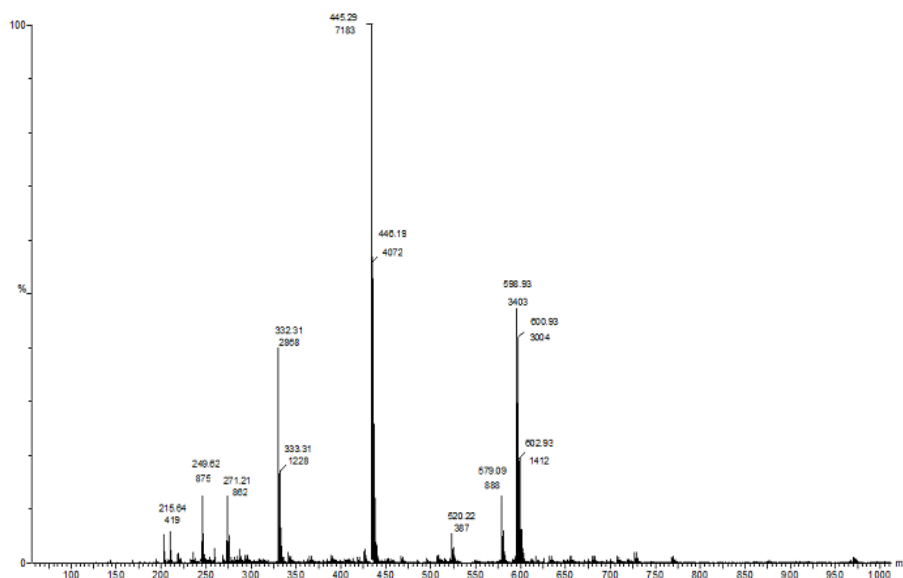


Figure 3: Mass spectrum of Co(II) LH complex.

CONCLUSION:

In the present study, the ligands and their Co(II), Cu(II) and Ni(II) metal complexes have been synthesized and characterized by elemental analysis, melting point, ^1H NMR, IR, UV-Vis spectra, and mass spectrometry measurements. According to the results obtained, all complexes are mononuclear and have been found to be four-coordinated. The mass spectra of ligands show molecular ion peaks in good agreement with the empirical formula suggested by elemental analysis. The activation energy calculated by the Freeman-Carroll and Sharp-Wentworth methods are in good agreement with each other. The thermal study revealed that complexes are thermally stable. The relative thermal stabilities of the complexes was in the order $\text{Co(II)} > \text{Ni(II)} > \text{Cu(II)} > \text{LH}$.

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