

Green & Efficient Synthesis, Spectroscopic Characterisation of Tridentate Schiff Base and its Metal Complexes

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Abstract:

Schiff base and its metal complexes have been synthesized by both Conventional Thermal Irradiation Technique (CTIT) as well as Micro Wave Thermal Irradiation Technique (MWTIT). later technique was found to be simple, quick, green, synthetic, pollution free and economically viable as compare to traditional one. This new green protocol involves one pot approach. Schiff base is prepared from the condensation of Anthranilic acid and Salicylaldehyde but in microwave assisted condensation in the absence of solvent is found a more efficient process to form a potentially tridentate Schiff base in solid state. This compound is versatile in forming well-defined complexes with transition metal (II) chloride. Schiff base ligand and its metal complexes have been characterized using various physicochemical techniques viz; elemental analysis, molecular weight determination, IR,¹HNMR, magnetic susceptibility measurement, molar conductance and ESR analysis. The analytical and spectral data showed that ligand acts as neutral tridentate with ONO donor sequence towards the transition metal(II) ions forming complexes of type [MLX_n] where M =Mn(II),Fe(II),Co(II),Ni(II),Cu(II), Zn(II),Cd(II) and Hg(II),L=C₁₄H₉NO₃,X= H₂O and n =2-3.The complexes possessed 1:1 metal-ligand ratio. ESR studies of Mn (II) complex explored the square pyramidal shape whereas other exist in octahedral and tetrahedral crystalline form.

Keywords: ESR study, Schiff base, green, spectral analysis, tridentate ligand.

Introduction

Schiff bases^[1] are the compounds containing azomethine group (–HC=N–) formed by condensation of primary amine and carbonyl compounds, also known as imine, aldimines or ketimines, first reported by Hugo Schiff^[2]. Schiff bases are prepared under acid or base catalysis or with heat by the conventional method. It can vary from noncyclic to cyclic and from unidentate to polydentate with different properties and structure according to the experimental conditions employed. The Schiff bases are crystalline^[3], these are feebly basic but at least form insoluble salts with strong acids. Schiff bases are readily hydrolyzed by aqueous acid to give back the amine and aldehyde. They can be reduced and may be used in preparation of secondary amine. Schiff base ligands are considered privileged ligands because they are easily prepared by a simple one-pot condensation^[4] with or without using solvent. Schiff bases have been widely used in many fields^[5] e.g. analytical, inorganic as monodentate to polydentate ligands in the area of coordination chemistry and drug synthesis. Schiff bases are utilized as starting materials in the synthesis of industrial compounds^[6], as anticorrosion agent and in catalytic activity^[7]. Some Schiff bases have also been used as perfumes, as dyes^[8], as fibers and as polymers. Many Schiff bases have been synthesized and characterized for their structural, isomeric, crystallographic and electronic importance. Due to these characteristics properties, Schiff bases have been used as materials for energy transfer^[9], materials for non linear optical response^[10], useful in telecommunication, in laser optics, optical data processing, in solar cells, in magnetic resonance imaging (MRI)^[11] and in nano level detection of metals^[12]. Metal complexes of Schiff bases are extensively studied due to synthetic flexibility^[13], selectivity^[14] and sensitivity towards a variety of metal atoms. They are found useful

in medicine as antibiotics ^[15], as an anti-inflammatory agent, as anti-HIV ^[16] and as anti-tumor agent ^[17]. Literature is abundant on physicochemical properties ^[18] of various Schiff bases and their complexes. Consequently, findings on Schiff base ligand (derived from the condensation of Anthranilic acid and Salicylaldehyde) and its metal(II) complexes with transition metal chloride are presented with the object of gaining more information about their nature of coordination, related structure, spectral study and antimicrobial properties.

Experimental

Materials and Methodology

All chemicals and reagents used for the preparation of ligand and complexes were commercial products (Sigma-Aldrich or Fluka) and were used without further purification. Solvents used for reactions were purified and dried by standard procedures ^[19]. Elemental analysis (carbon, hydrogen, nitrogen and oxygen) were determined with an Elemental CHNO analyzer, Vario EL (III), Carlo Erba 1108 model. Molecular weight determination was carried out with Shimadzu K-7000. Melting points were recorded out using an electro-thermal apparatus and are uncorrected. Conductivity measurements were made with DMSO solutions using a PW 9526 digital conductivity meter and room temperature magnetic moments were measured with a magnetic susceptibility balance (Johnson Matthey Catalytic System division). Infrared spectroscopic data (KBr disc, 4000-400 cm^{-1}) were obtained on a Perkin-Elmer FT-IR (model RXI) spectrophotometer. The ¹H-NMR spectroscopic data were made on a Bruker DRX-300, 400MHz in CDCl_3 and chemical shifts were indicated in ppm relative to tetramethylsilane. ESR was measured on Varian model E-112.

Synthesis of Schiff base and its metal (II) complexes

Conventional Thermal Irradiation Technique and Microwave Irradiation Technique were used and reported in this paper.

Synthesis using CTIT

Schiff base ligand (H_2SB_1) was prepared ^[20, 21] by condensation of respective carbonyl compound with primary amine. The mixture of two was dissolved in minimum quantity of ethyl alcohol and refluxed for 4 hrs. The resulting solution on cooling provided colored precipitate of Schiff base. The precipitate was filtered and recrystallised in ethanol and dried in air. The final product was subjected to physicochemical examination.

Synthesis of Schiff base metal complexes with d block elements

A solution of metal (II) chloride in DMF was mixed with the Schiff base (H_2SB_1) in (1:1) Metal: Ligand molar ratio, respectively. The mixture was refluxed for 1.5 to 5.0 hrs. then poured into ice cold water, a colored solid separate out. The product was filtered, washed with ether, recrystallized from DMSO and DMF and dried over anhydrous CaCl_2 in vacuum at room temperature. The yield was found 51-78% in all the complexes, do not display sharp melting points and undergo decomposition in the temperature range of 220–255°C, were slightly to fairly soluble in common organic solvents viz; alcohol, ether, benzene, hexane, DMF and DMSO but totally insoluble in water.

Synthesis using MWTIT

Schiff base was synthesized ^[22, 23] by equimolar mixture of respective carbonyl compound with amino compound. The calculated quantities (1:1) of both mixed and tarturated dry in glass mortar for one to two hours. Finely ground and evenly mixed components were then subjected to the microwave. Reaction got completed in just few seconds.

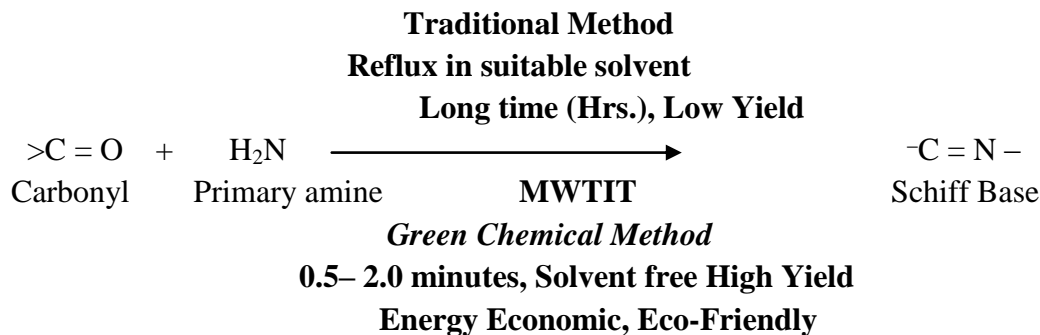
Synthesis of Schiff base metal complexes with d-block elements

The metal (II) chlorides of d block and Schiff base ligand were mixed in proper molar ratio, crushed, tarturated dry and subjected to microwave irradiation for few seconds to few minutes in a domestic microwave oven without using catalyst and solvent. The Schiff base metal complexes were obtained in higher yield (75.66 to 92%) as compared to CTIT.

Comparative study:

A comparative study regarding yield and reaction period is as follows:

CTIT



Result and discussion

The preparation of a novel Schiff base ligand and its complexes with the appropriate transition metal(II)chloride, such as Mn,Fe,Co,Ni,Cu,Zn,Cd and Hg by both routes (conventional and microwave) were reported but latter method was easy early, economic, and eco-friendly over conventional one. The Schiff base ligand and its metal (II) complexes were displayed good stability in solid state at room temperature and which were not shown by metal (III) complexes thus not reported. These compounds were characterized by the usual methods: elemental analysis, molar conductance, magnetic moment FTIR, 1H -NMR, and ESR spectral analysis. On the basis of micro analytical data, metal complexes under investigation may be represented by formula MLX_n , where M stands for metal ion, L for ligand- Schiff base, X for the water molecule and n is number of water molecule. The Schiff base ligand was expected to act as a tridentate ligand, the possible coordination sites being imine nitrogen, the oxygen atom of the deprotonated salicylaldehydic group and the oxygen atom of the deprotonated carbonylic group (Figure 1). A study and comparison of the IR spectra of the ligand and its metal complexes imply that the Schiff base behaves as a dibasic tridentate ligand with ONO donor sites. Various attempts such as crystallization using mixtures of solvents, low-temperature crystallization were unsuccessful to obtain a single crystal suitable for X-ray crystallography.

Schematic representation of Schiff base ligand and its metal complex formation along with comparative study in terms of reaction period, yield, elemental analysis and some physical data of the compounds are reproduced in (Table1).All the compounds were nonhygroscopic, colored solids, insoluble in water but soluble in all common organic solvents ^[24].

Molar conductivity

Molar conductivity of the freshly prepared solution of the complexes in DMSO showed conductance value in the range of 4.12- 4.77 $\text{ohm}^{-1} \text{cm}^2 \text{mole}^{-1}$ which indicated that the complexes were neutral suggested ^[25] 1:1 electrolytic nature for the transition metal complexes.

Magnetic susceptibility

The observed magnetic moment of Fe (II) complex was 5.3 B.M., thus the complex was in octahedral geometry involving sp^3d^2 hybridization. The magnetic moment of Co (II) complex was measured lies in 4.2

B.M. (normal range for octahedral Co (II) complexes is 4.2 – 5.2 B.M. which was indicative of octahedral geometry. The Ni(II) complex reported here in was high spin with magnetic moment value 3.24 B.M. at room temperature, which was the normal range reported for octahedral Ni(II) complexes ($\mu_{\text{eff}} = 2.9 - 3.3$ B.M.). This indicated that the Ni (II) complex was six-coordinated and probably octahedral. Generally it was observed that in Cu (II) ion, d-d transitions are normally close in energy. The magnetic moment of 1.93 to 2.07 B.M. falls within the range, normally observed for octahedral Cu (II) complexes. The magnetic moment of $\text{H}_2\text{SB}_1 - \text{Cu}$ (II) chelate was found to be 1.98 B.M., which suggested a high-spin distorted octahedral or tetragonal geometry in terms of John-Teller effect for these complexes. This structure is also evident from analytical data of the complex. The complexes of Zn (II), Cd (II) and Hg (II) of H_2SB_1 were diamagnetic. In analogy with those described for metal (II) complexes containing Schiff base, we proposed tetrahedral geometry for Zn (II), Cd (II) and Hg (II) chelates.

IR spectra and mode of bonding

The IR data of Schiff base H_2SB_1 and its complexes are shown in (Table2). The spectra of the ligand and metal complexes were taken in the range of $4000-400\text{cm}^{-1}$ for confirmation of the complexation. The IR spectra of the complexes were compared with free ligand in order to determine the coordination sites that may be involved in chelation. There were some guide peaks in the spectra of the ligands which were helpful in achieving this goal. The spectral data of the ligand revealed a strong absorption band at 1622 cm^{-1} due to C=N stretching indicating the conformation of azomethine group which was shifted to 1540cm^{-1} , the lower side in $\text{H}_2\text{SB}_1\text{-Ni}$ complex. This supported the fact that ligand coordinate to metal ion through the nitrogen of C=N group. The band at 1687 cm^{-1} was attributed to $\nu(\text{C}=\text{O})$ carboxylic which was shifted to lower frequency 1622 cm^{-1} in case of $\text{H}_2\text{SB}_1\text{-Ni}$ complex, revealed the confirmation of involvement of carboxylic-OH bond in complexation^[26]. The salicylaldehydic ν_{OH} was absent in complex further confirmed the involvement in complexation. It was determined that the strong bands were observed in ligand at 1596 due to $\nu(\text{C}=\text{C})$ stretching^[27] which remained almost unaffected on coordination.

The two broad bands at 3572 cm^{-1} and 3383 cm^{-1} were observed for ligand H_2SB_1 and $\text{H}_2\text{SB}_1\text{-Ni}$ complex due to lattice water. The bands at 423 cm^{-1} for $\nu_{\text{M-O}}$, 656 cm^{-1} for $\nu_{\text{M-O}}$ and 754 cm^{-1} for $\nu_{\text{M-N}}$ were present in metal complex and absent in parent ligand indicating the formation of bonds with central metal ion Ni. It was concluded that the Schiff base (H_2SB_1) behaved as a neutral tridentate ligand, coordinated to the metal ions via azomethine- N, carboxylic-H, salicylaldehyde-O. (Figure 4)

¹HNMR spectra

The ¹HNMR spectrum of the Schiff base and its metal complexes were recorded in DMSO-d₆ using TMS as internal standard and data are given in (Table3).

¹H-NMR spectrum of Schiff base was shown the following signals; $\delta\text{H}(400\text{MHz}, \text{DMSO-d}_6)$: H_2SB_1 : ¹HNMR (δppm) 10.2655 (S,H,carboxylic), 8.8586 (br, H, salicylaldehyde OH), 6.4723–7.8594 (S, 4H, aromatic and 4H salicylaldehyde), 3.3647 (S,H, azomethine), 2.5069 (S, CH₃ of DMSO)

The ¹HNMR spectra of the Schiff base ligand exhibited strong resonance peak^[28] at 10.26 and 8.85 ppm due to proton of the carboxylic group and OH group present on salicylaldehyde respectively. The appearance of strong peak at 3.36 ppm was assignable to proton of azomethine group which was absent in metal complexes. This finding may be taken as evidence for the participation of ($-\text{HC}=\text{N}-$) group in coordination to the metal ions. The sharp phenyl multiplates (4H) and salicylaldehyde multiplates (4H) were observed in the range of 6.47-7.85 ppm which remained unchanged as also observed in all the complexes indicating the noninvolvement of these protons during complexation. Moreover, signals due to protons of carboxylic group and salicylaldehydic group were not found revealed their participation in complex formation. A study and comparison imply that

Schiff base behaves as dibasic tridentate ligand and each metal ion is coordinated through the deprotonated oxygen atom of carboxylic group, deprotonated oxygen atom of salicylaldehydic group and nitrogen atom of azomethine group (Figure 5).

Electron spin resonance spectra

The ESR spectra of the complex provide valuable information on the metal environment. The ESR spectra of Mn (II) complex with H₂SB₁ was recorded as polycrystalline sample on X band at frequency 9.1GHz at room temperature (CLNT low nitrogen temperature, if it was required) under the magnetic field strength 3000 Gauss. The g value was calculated by Kivelson method = $(g_{\parallel} - 2)/(g_{\perp} - 2)$. The analysis of the spectra showed G value was less than one ($G < 1$) which confirms considerable exchange interaction in solid complexes. The analysis of spectra displayed g_{\parallel} (5.65) and g_{\perp} (5.26) indicate the unpaired electron was localized in $d_{x^2-y^2}$ giving $g_{\parallel} > g_{\perp} > 2$ ($d_{xy}, d_{yz}, d_{zx}, d_{x^2-y^2}$ & d_y^2) of the Mn(II) ion (Figure 6).

Conclusion

The synthesis of a Schiff base ligand and its complexes with the transition metal (II) chloride, such as Mn, Fe, Co, Ni, Cu, Zn, Cd and Hg by both routes (conventional, using suitable solvent and microwave with no solvent) were presented for the sake of comparison. Microwave Thermal Irradiation Technique (MWTIT) was used for providing the desired amount of activation energy in the synthesis and was found to be green organic synthesis without solvent to avoid waste, be atom efficient, clean production and cheap with greater yield as compared to Conventional Technique. While performing experiment, so many drawbacks were observed such as unsatisfactory yields, cumbersome product isolation procedure, release of chemical waste to environment making it polluted and technical intricacy. Moreover the main disadvantage of this protocol is longer reaction time, more energy consumption and hazardous chemical waste sunk from laboratories. To overcome these problems simple and ecofriendly –benign transformation using energy from microwave field under dry condition, the Schiff base ligand and its metal complexes were prepared by simply tarturating the reactants (1:1 molar ratio) in using glass paste. Method is found fast; efficient with better yield of products thus this green protocol can be commonly applied to overcome the traditional one.

These compounds were characterized by elemental analysis, molar conductance, magnetic moment, FTIR, ¹H-NMR and ESR spectral analysis. Spectral and analytical data of the ligand imply that the Schiff base behaves as a dibasic tridentate ligand with ONO donor sites and resultant data revealed that the metal (II) (Fe, Co, Ni & Cu) complexes have octahedral structure. The ESR spectra confirmed square pyramidal geometry for the Mn (II) complex.

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Table I Characterisation of Schiff Base [H₂SB₁] and its Metal Complexes

S. No.	M-L	Synthesis Technique		Physical State (Colour)	MP (°C)	Mol.WtObsd. (Calcd.)	Elemental Analysis Obsd. (Calcd.) %					μ_{eff} B.M. at 308K	M (ohm ⁻¹ cm ² mole ⁻¹)	n ^o	B	Molecular Formula
		CTIT	MWTIT				M	C	H	N	O					
		Yield %	Yield %													
		(Reaction time, hrs.)	(Reaction time, Sec.)													
1	H ₂ SB ₁	63.44%	75.66	Solid	221	243.10	-	68.65	4.15	5.82	19.80	-	-	-	-	(C ₁₄ H ₁₁ NO ₃)
		(4.00)	(30.00)	(Crimson)		(241.00)	-	(69.68)	(4.56)	(5.80)	(19.91)					
2	Mn-SB ₁	63%	90%	Green	225	329.11	15.11	47.11	2.54	3.99	13.62	5.11	3.86	3	sp ³ d	[Mn(C ₁₄ H ₉ NO ₃).(H ₂ O) ₂]
		(2.00)	(4.00)	Greenish yellow		329	15.80	48.27	2.58	4.02	13.79					
3	Fe-SB ₁	64%	80%	Solid	220	347.25	15.78	48.11	4.21	4.00	2.71	5.32	4.12	4	sp ³ d ²	[Fe(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(3.00)	(2.00)	(Brick)		(348.85)	(16.00)	(48.15)	(4.29)	(4.01)	(2.75)					
4	Co-SB ₁	62%	79%	Solid	236	350.88	16.34	47.64	4.16	3.88	2.71	4.19	4.37	3	sp ³ d ²	[Co(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(4.50)	(6.50)	(Brick)		(351.93)	(16.74)	(47.73)	(4.26)	(3.97)	(2.72)					
5	Ni-SB ₁	65%	83%	Solid	238	348.66	15.66	48.01	4.23	4.00	2.73	3.26	4.77	2	sp ³ d ²	[Ni(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(2.50)	(2.00)	(Lem.Yellow)		(348.69)	(15.97)	(48.18)	(4.30)	(4.01)	(2.75)					
6	Cu-SB ₁	78%	92%	Solid	250	355.58	17.61	47.09	4.11	3.82	2.61	1.98	4.19	1	sp ³ d ²	[Cu(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(3.00)	(1.00)	(Dark Green)		(356.55)	(17.82)	(47.11)	(4.20)	(3.92)	(2.69)					
7	Zn-SB ₁	71%	91%	Solid	252	357.78	18.11	46.74	4.18	3.81	2.09	-	-	0	sp ³	[Zn(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(5.00)	(1.00)	(Peach)		(358.39)	(18.24)	(46.87)	(4.18)	(3.90)	(2.67)					
8	Cd-SB ₁	65%	87%	Solid	255	404.87	26.67	41.34	3.66	3.41	2.33	-	-	0	sp ³	[Cd(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(1.50)	(1.10)	(Crimson)		(405.41)	(27.72)	(41.43)	(3.69)	(3.45)	(2.36)					
9	Hg-SB ₁	66%	84%	Solid	232	492.98	40.53	34.00	3.01	2.61	1.91	-	-	0	sp ³	[Hg(C ₁₄ H ₉ NO ₃).(H ₂ O) ₃]
		(2.50)	(1.50)	(Yellow)		(493.59)	(40.63)	(34.03)	(3.03)	(2.83)	(1.94)					

n^o= No. of Unpaired Electrons

B = Possible Bond Type

Table 2 IR data (KBr pellet cm^{-1}) of H_2SB_1 and its Metal Complexes

M-L	Molecular Formula	$\nu_{C=N}$	ν_{C-O}	ν_{H_2O}	ν_{M-N}	$\nu_{(M-O)}$	$\nu_{(M-O)}$
H₂SB₁	(C ₁₄ H ₁₁ NO ₃)	1622.1s	1687.3s	3572.6w	-	-	-
Mn-SB₁	[Mn(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1545.94s	1615.45s	3359.42br	763.32s	634.07s	574.8s
Fe-SB₁	[Fe(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1615 s	1680.08m	3470.12br	763.23s	428.11m	606.01m
Co-SB₁	[Co(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1620 s	1677.22s	3473.21br	768.33s	430.21m	605.02m
Ni-SB₁	[Ni(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1540.23m	1622.49m	3383.84br	754.44s	423.40m	656.7s
Cu-SB₁	[Cu(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1606.23 s	1789.20m	3446.03br	720.01s	455.01m	620.11.2m
Zn-SB₁	[Zn(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1620.34 s	1675.02m	3470.04br	767m	430.10s	615.20s
Cd-SB₁	[Cd(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1587.8s	1609.96m	3418.61br	757.31s	450.01m	679.09m
Hg-SB₁	[Hg(C ₁₄ H ₉ NO ₃). ₃ (H ₂ O) ₃]	1619.12 s	1670.45m	3471.51br	761.11s	425.52m	614.23s

s= strong, w=weak, m= medium, br=broad

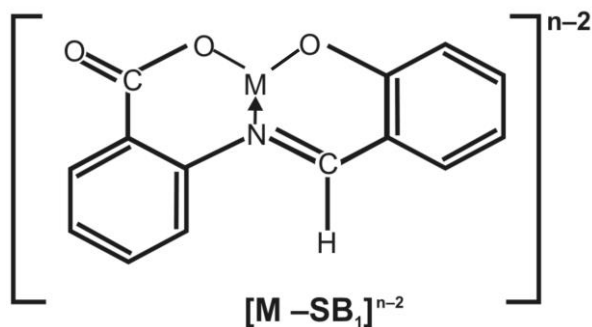
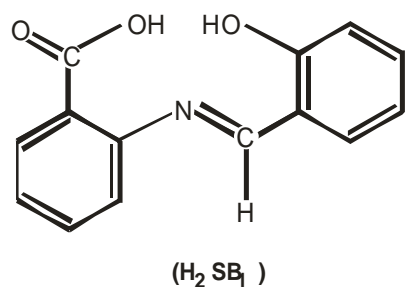
Table3 ¹HNMR of H_2SB_1 and its $\text{SB}_1\text{.Cd}$ Complex

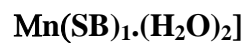
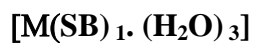
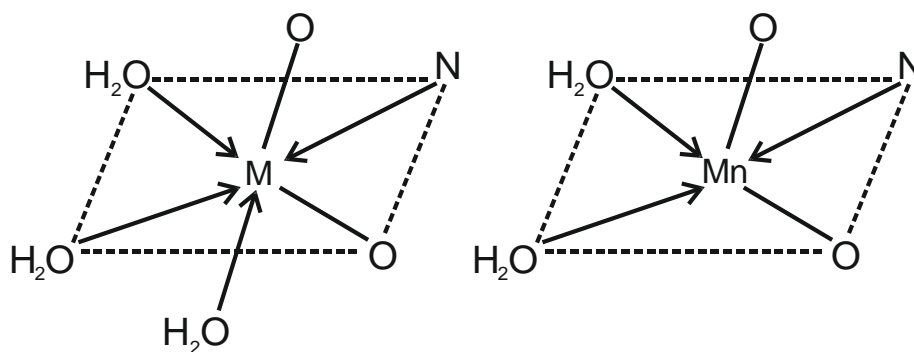
S.No.	Chemical Shift, (δ) ppm		Assignment
	H_2SB_1	$\text{SB}_1\text{.Cd}$	
1	10.2655	Absent	S,H,carboxylic
2	8.8586	Absent	Br, H, salicylaldehyde OH
3	6.4723-7.8594	6.065 -7.227	S, 4H, aromatic and 4H, salicylaldehyde
4	3.3647	3.337	S,H, azomethine

Table 4 Antimicrobial activity of the Schiff Base (H₂SB₁) and its metal complexes as percentage (%) zone of inhibition with different dilution

S.No.	Schiff Base and its Complexes	<i>Staphylococicoagulase (Gram+)</i>			<i>Escherichia coli(Gram-)</i>		
		Negative Control :Vancamycin (12mm)			Positive Control :Ceflaxomycin; (13mm)		
		50ppm	100ppm	200ppm	50ppm	100ppm	200ppm
1	H ₂ SB ₁	1mm	2mm	3mm	-	-	-
2	SB ₁ -Mn	2mm	4mm	6mm	15mm	22mm	25mm
3	SB ₁ -Fe	6mm	6mm	6mm	5mm	7mm	10mm
4	SB ₁ -Co	8mm	10mm	13mm	7mm	10mm	15mm
5	SB ₁ -Ni	5mm	8mm	11mm	6mm	7mm	20mm
6	SB ₁ -Cu	4mm	7mm	10mm	15mm	17mm	20mm
7	SB ₁ -Zn	8mm	11mm	15mm	10mm	13mm	15mm
8	SB ₁ -Cd	30mm	35mm	40mm	18mm	20mm	23mm
9	SB ₁ -Hg	7mm	10mm	13mm	6mm	8mm	13mm

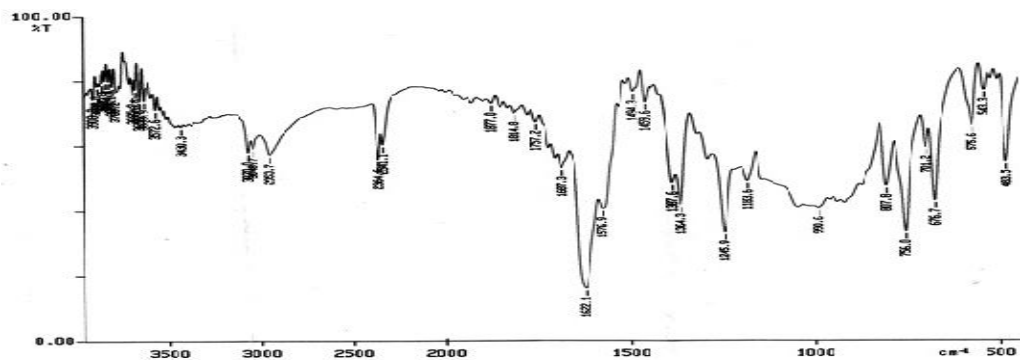
(McFarland turbidity standard: The McFarland standard is used to adjust the turbidity of the inoculum for the susceptibility test).

**Figure 1 Structure of Schiff Base****Figure 2 Structure of Schiff Base Metal complex (n=2)**

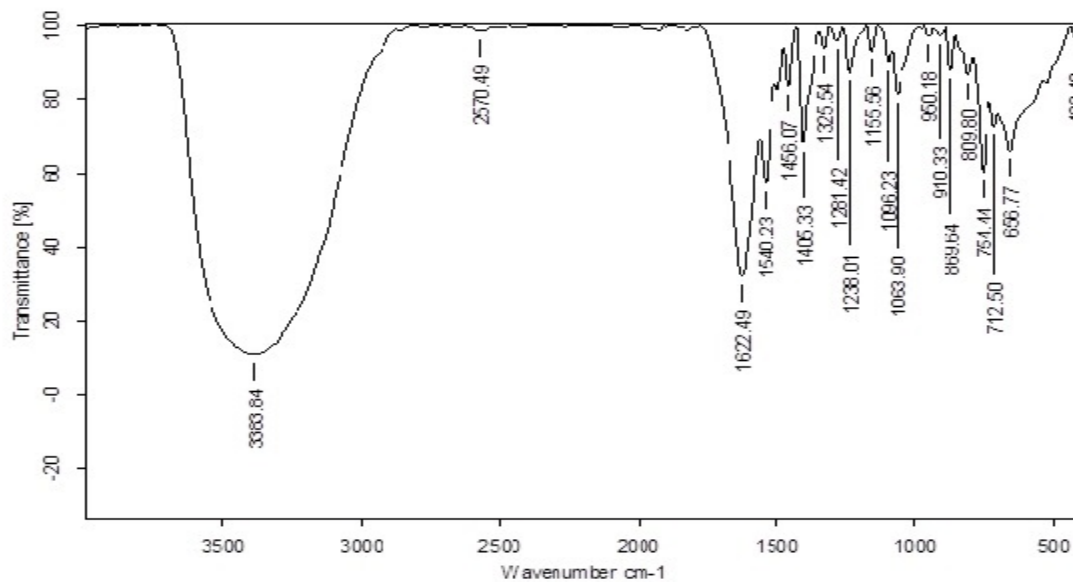


Where, M = Mn (II), Fe (II), Co (II), Ni (II), Cu (II),

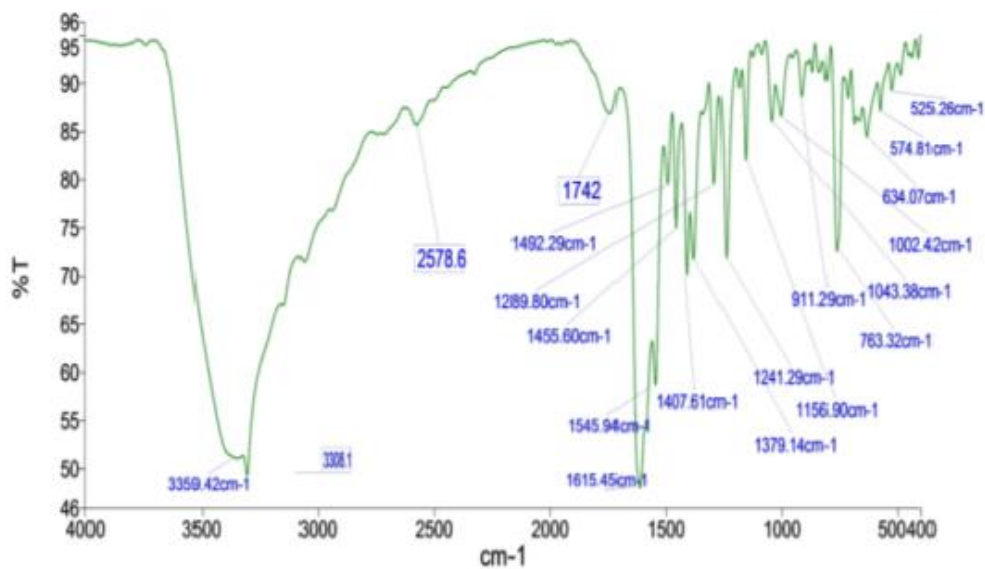
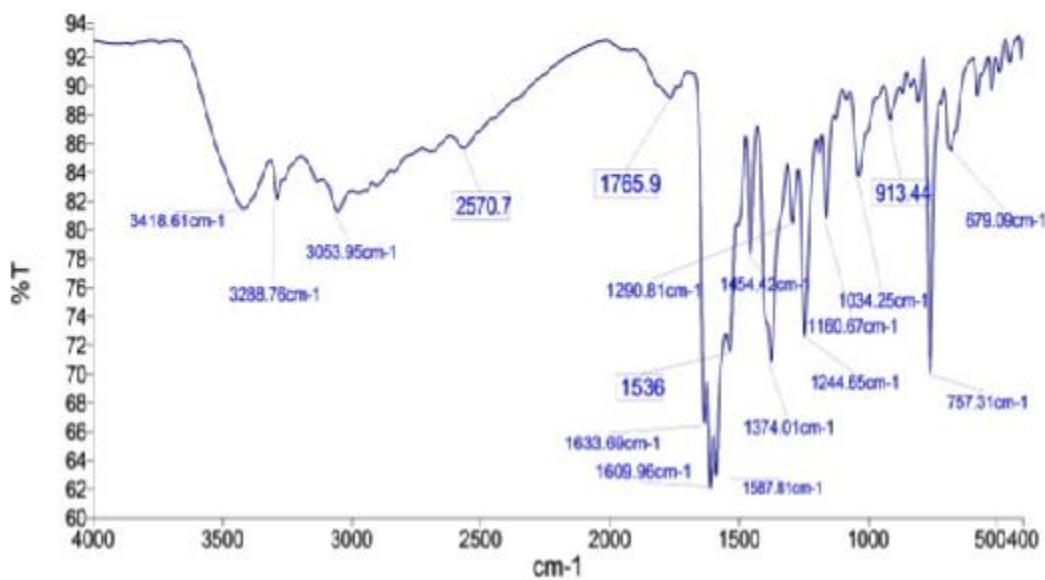
Figure 3 Structural Formula of Schiff Base Metal (II) Complex



H_2SB_1



SB_1-Ni

SB₁-MnSB₁-CdFigure 4 IR Spectra of H₂SB₁ & its metal complexes

