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# Preparation and Properties of binuclear Schiff Base Complexes of Cu (II) Co(II) and Ni(II) Inter-complex Reaction

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**ABSTRACT:** Homo and hetero binuclear Schiff base complexes of Ni(II), Cu (II) and Co (II) were prepared by inter-complex reaction between the corresponding metal complexes of 2-hydroxy-1-naphthaldehyde and 2-amino-3-hydroxy pyridine. The complexes were characterized by elemental analysis and estimation of metals. Thermal behavior of the complexes was studied by TG-DTA analysis. Structures of the complexes were elucidated by spectroscopic methods like, infrared spectroscopy, UV-visible spectroscopy, mass spectrometry and <sup>1</sup>H NMR spectroscopy. The powder X-ray diffraction study suggested crystalline nature of the complexes with tetragonal geometry. Magnetic moments and electronic spectra reveal tetrahedral structure of the complexes. Antibacterial activity of complexes were studied against Gram-positive bacteria, *Staphylococcus aureus*, *Bacillus subtilis* and Gram-negative bacteria, *Salmonella typhi*, *Escherichia coli* by agar cup method. Their antifungal activity was also tested against *Aspergillus niger*, *Penicillium chrysogenum*, *Fusarium moniliforme* and *Aspergillus flavus* by poison plate method using potato dextrose agar medium at one percent concentration. All complexes show considerable antimicrobial activity.

**KEYWORDS:** Schiff base, inter-complex reaction, binuclear complex, biological activity

## I. INTRODUCTION

Mixed metal complexes differ from traditional complexes in the sense that they are having at least two different or same metals associated with two different ligands (metal organic ligands) the presence of more than one type of ligands in a complex increases chances of variation in properties expected for the complex. This makes the researcher interested in the synthesis of mixed metal complexes with varying properties. [1] Synthesis and characterization of mixed metal complexes is gaining importance day by day. The increased interest in this research area has motivated many researchers to get involved in this field. In recent years many publications are devoted to synthesis and characterization of mixed metal as well as ligands complexes. [2-6] The Schiff base complexes were also used as drugs and they possess a wide variety of antimicrobial activity against bacteria, fungi and it also inhibits the growth of certain type of tumors. [7-8] The complexes formed by coordination with metal ions, have the tendency to coordinate further or react with other complexes, then they may act as metal organic ligand (MOL). The donor atoms are unable to coordinate with the same metal ions due to steric factors. This unutilized functionality is drawn on another metal ion forming poly nuclear complex [9-10]

Here a complex containing some unutilized functionality in ligand is considered as a ligand and named as metal organic ligand (MOL). This (MOL) when allowed to react with metal ions result in the formation of mixed metal complexes. We report here, a novel approach of synthesizing mixed metal complexes. It has been hypothesized that the coordinated ligands of two metal chelates can be reacted to obtain a new metal chelate. In the present work, we have allowed two such complexes under the conditions that permit coordinated NH<sub>2</sub> to react with the coordinated CHO group. Here an ionic bonds of the precursor do not dissociate and metal-ligand bonding in both the complexes remains intact [11]. Due to the reaction between coordinated amine and aldehyde groups, Schiff bases were formed. The imine nitrogen of the Schiff base was allowed to coordinate with the metal nearby while the deficiency created at the metal ion on aldehyde end was sufficed by aqua-ligands liberated during imine formation. The resultant binuclear complex thus has one of the metal ions in di aqua form. When the metal ion in the reacting complexes was different, the resultant complexes were mixed metal complexes [12].

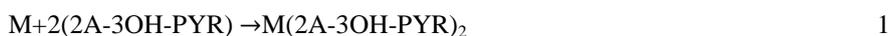
## II. MATERIALS AND METHOD

**Reagents:** 2-amino 3-hydroxy pyridine and 3-ethoxy salicylaldehyde (>99.0%) were purchased from S.D. Fine Chemicals. Nickel acetate, copper acetate, sodium hydroxide and solvents (>99.0%) were purchased from E-Merck Ltd, Mumbai (India). The purification was done according to the needs through known procedures.

**Measurements:** Elemental analysis (C, H, N & O) was done using Perkin Elmer, series II, 2400 CHNS/O Analyzer. The metal content of the complexes were determined by EDTA titration method after decomposition of the metal complexes with an acid mixture of HClO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> (1:1.5:2.5) in case of Fe<sub>2</sub>(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>. The amount of Fe(II) from homo binuclear complex of Fe(II) viz Fe<sub>2</sub>(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> was determined by EDTA titration method. FeZn(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> was done by separating the iron from zinc. Solution containing a mixture of metal ions, before precipitating iron as hydroxide, add 5grms of NH<sub>4</sub>Cl to retain zinc in the solution. Now estimate the iron as iron oxide gravimetrically. Reserve the filtrate and washings for volumetric estimation of zinc by titrating against standard EDTA solution volumetrically. In the same manner separation and estimation metals of FeMn(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> can be done by separating iron as iron oxide gravimetrically and Mn(II) volumetrically by titrating against standard EDTA solution volumetrically. All chemicals used were of analytical grade and used without purification. All metal salts were purchased from SD fine chemicals. Elemental analysis (C, H, N, O) were performed on Perkin Elmer-2400. IR spectra were recorded on FTIR spectrophotometer model RZXC Perkin-Elmer in the range (4000-400 cm<sup>-1</sup>), <sup>1</sup>H NMR spectra were recorded on Bruker Avance II at 400 MHz using tetramethylsilane as an internal standard. Electronic spectra was recorded on the Shimadzu 1800 spectrophotometer using DMSO as a solvent. Mass spectra were recorded on Waters, Q-TOF Micro Mass (LC-MS). Magnetic data at room temperature were collected on Gouy balance. Mercury (II) tetrathiocyanate cobalt acetate was used as a calibrant. Diamagnetic contributions were calculated for each compound by Pascal's constants. TGA/DTA analysis was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000. Heating rate was 10°/min. x-ray diffractogram was scanned on Bruker AXS D<sub>s</sub>.

### Synthesis of Metal Complexes:

The method used in the synthesis of metal complexes consists of following three steps. In the first step, 2-amino-3-hydroxy pyridine (2A-3OH-PYR), (0.404gm) in absolute alcohol (~20mL) was prepared and a solution of Copper /Cobalt / Nickel acetates (0.497g/0.399g) in rectified spirit (20mL), were mixed, stirred for an hour to obtain a four coordinated complex, M(2A-3OH-PYR)<sub>2</sub> in solution as shown in equation-1,



In the second step, 3-ethoxy salicylaldehyde (3E-SAL), (0.665 g) in absolute alcohol (~20ml) was prepared and a solution of Nickel/Copper /Cobalt acetates (0.5g, 0.1m) in rectified spirit (~20ml), were mixed and stirred for an hour to obtain a four coordinated complex, M'(3E-SAL)<sub>2</sub> in solution. The reaction is shown in equation 2.



In third step, a solution of M (2A-3OH-PYR)<sub>2</sub> was added to the refluxing solution of M' (3E-SAL)<sub>2</sub>. The reaction mixture was refluxed for 6-hours in a water bath to obtain the product under slightly alkaline condition created by sodium hydroxide. The precipitate was then filtered, washed with ethanol and dried over fused CaCl<sub>2</sub>. The third step of the reaction is depicted in equation 3.



All complexes were prepared by the above discussed method. The heterodinuclear complex, whereas homobinuclear complex, Cu<sub>2</sub>(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>, CuCo(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> and CuNi(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> were obtained when M=Cu and M'=Co (II), Ni(II) respectively in heterodinuclear complexes and M & M'=Cu in mononuclear complex. The melting points of all the complexes were found to be higher than 300°C.

## III. RESULTS AND DISCUSSION

**IR Spectra :** The IR Spectra of reactant complexes and dinuclear complexes displayed some similarities and dissimilarities, Significant IR bands are shown in Table 1. The spectra of the reactant complex M(2H-3AP)<sub>2</sub> showed a strong absorption at 1551 cm<sup>-1</sup> frequency<sup>1</sup> which was assigned to coupled vibrations of NH<sub>2</sub> bending and stretching



[13] absorptions at  $3330\text{ cm}^{-1}$  were attributed to  $\text{NH}_2$  asymmetric and symmetric stretching frequency respectively. A weak band at  $556\text{ cm}^{-1}$  was observed in the complex which was assigned to the M-N stretching .

IR spectra of reactant complex  $\text{M}^+(3\text{E-S})_2$  exhibited a broad band and strong peak at  $1530\text{ cm}^{-1}$  which was assigned to C=O stretching in the complex. A weak band at  $456\text{ cm}^{-1}$  observed in the spectra was due to M-O stretching frequency. Both the complexes showed a band in the region of  $3330\text{ cm}^{-1}$  &  $3365\text{ cm}^{-1}$  arising due to aromatic ring vibrations the spectra of both the reactant complexes did not show a broad band in the region of  $3400\text{ cm}^{-1}$  which indicated the absence of any coordinated water molecule.

In the spectra of resulting binuclear complexes viz  $\text{MM}^+(\text{SB})_2(\text{H}_2\text{O})_2$  peak due to C=O stretching ( $1530\text{ cm}^{-1}$ )  $\text{NH}_2$  bending and  $\text{NH}_2$  stretching ( $1551\text{ cm}^{-1}$ ) was found to be absent .New stronger bonds appearing at  $547\text{-}582\text{ cm}^{-1}$  and  $401\text{-}460\text{ cm}^{-1}$  were assigned to M-O and M-N stretching frequencies. A broad band in the region  $3400$  shows presence of two coordinated water molecules and a sharp and strong peak between  $1600\text{-}1597\text{ cm}^{-1}$  which may be attributed to C=N stretching was in accordance with the proposed structure of the complex.

**Table 1: FT-IR Spectral frequencies of Complexes**

System	VC=N $\text{cm}^{-1}$	VO-H $\text{cm}^{-1}$	VM-O $\text{cm}^{-1}$	VM-N $\text{cm}^{-1}$
$\text{M}^+(3\text{E-S})_2$			556	
$\text{M}(3\text{H-2AP})_2$			594	418
$\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$	1608	3411	539	466
$\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$	1608	3456	539	468
$\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$	1608	3434	540	418

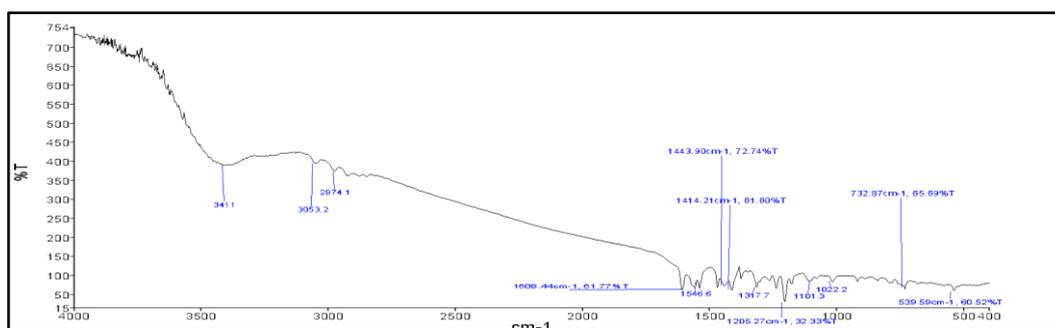


Fig :  $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$

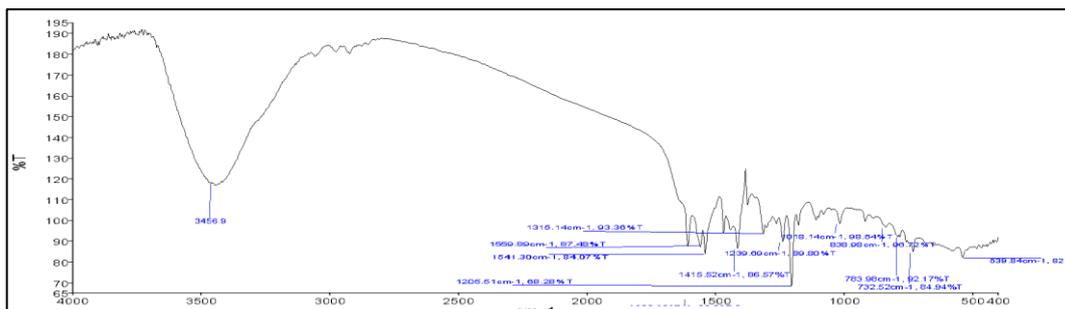


Fig :  $\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$

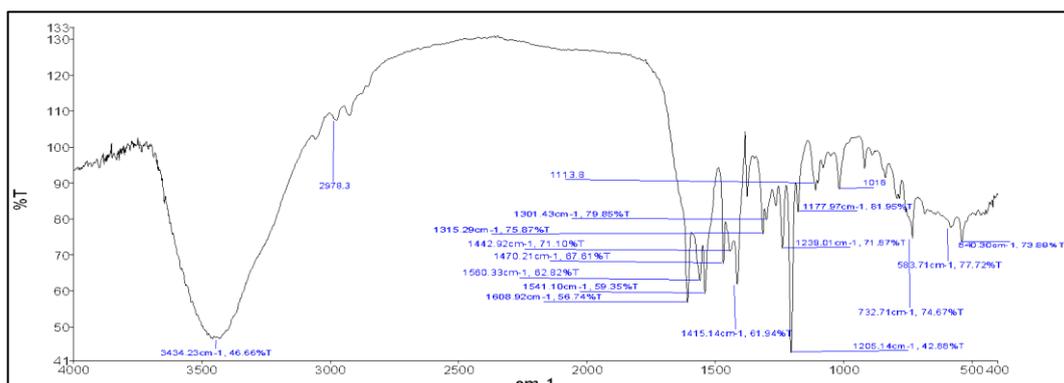


Fig : CuNi (SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>

**Electronic Spectra and Magnetic Studies**

All the complexes showed absorption peaks in the near UV region and these high intensity bands were due to  $\pi \rightarrow \pi^*$  transition in the aromatic group of ligand. The spectra of the homodinuclear complex Cu<sub>2</sub>(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> is characterized by two weak bands at region, 22222 cm<sup>-1</sup> & 24509 cm<sup>-1</sup> assigned to 5T<sub>2g</sub>→3T<sub>1g</sub>, 4A<sub>2g</sub>→4T<sub>1g</sub> transitions respectively. The effective magnetic moment at room temperature for Cu<sub>2</sub>(SB)(H<sub>2</sub>O)<sub>2</sub> was found to be 2.21BM for each Cu(II) ion that was slightly higher than the suggested magnetic moments for the tetrahedral geometry of Copper[14]. The spectra of Hetero nuclear complexes complex CuCo(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> is characterized by two weak bands at region, 13,114 cm<sup>-1</sup> (762nm), 24,301 cm<sup>-1</sup> (411nm) assigned 3T<sub>2g</sub>(F) →3T<sub>2g</sub>(F), 6A<sub>1g</sub>→4A<sub>1g</sub>Eg (G) transitions respectively and charge transfer transitions indicating tetrahedral geometry around the metal ions. In the present investigation electronic spectra of the complex shows absorption peaks in the near ultra-violet region and these high intensity bands were due to  $\pi \rightarrow \pi^*$  transition in the aromatic group of ligand [15]. The electronic absorption spectra of CuNi (SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> complex, bands observed at 15,337 cm<sup>-1</sup> (652nm), 21,032 cm<sup>-1</sup> (475nm), assigned to 3T<sub>2g</sub> →3T<sub>1g</sub>, 3T<sub>2g</sub>(F)→3T<sub>1g</sub>(p), 4A<sub>2g</sub>→4T<sub>1g</sub> and charge transfer transition indicating tetrahedral geometry around the metal ions. The higher value of the effective magnetic moment suggests the presence of some ferromagnetic interaction at room temperature. On the basis of physico-chemical and spectral study, Following structure may be proposed for the complexes.

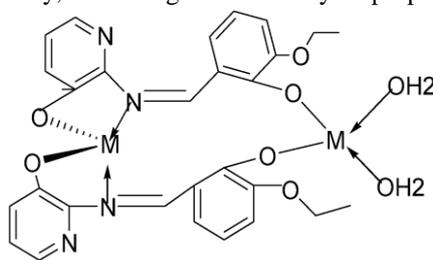


Fig-1 Proposed structure for the complexes

**Table2: Physicochemical and analytical data of metal complexes**

System	Mol. Wt g/mole	Color	% Yield	$\mu_{eff}$ per ion B.M.	Elemental Analysis % Found (Calculated)						
					C	H	N	O	Cu (II)	Ni (II)	Co (II)
Cu <sub>2</sub> (SB) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	664	Dark brown	80	2.29	41.00 (41.15)	2.20 (2.21)	6.66 (6.71)	17.60 (17.64)	17.50 (17.68)	18.64 (18.87)	
CuNi(SB) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	663	Yellowish Green	74	*	50.20 (50.30)	3.20 (3.29)	8.29 (8.37)	9.49 (9.57)	9.48 (9.51)	8.89 (8.87)	

$\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$	668	brown	72	*	49.49 (49.50)	2.80 (2.75)	8.00 (7.89)	9.50 (9.57)	8.79 (8.81)		9.45 (9.50)
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**Mass and <sup>1</sup>H-NMR Spectra of the Complexes:**

**Mass Spectra**

The molecular weights of all the binuclear complexes are exactly equal to that calculated theoretically from the proposed structures. These results are further supported by the conclusions drawn from the elemental analysis which agree with the molecular formula assigned to these complexes.

Formation of binuclear metal complexes and their structure is confirmed by <sup>1</sup>HNMR spectral study of representative metal complexes. The result obtained was used to interpret the proton environment and number of protons present in the sample. The <sup>1</sup>HNMR spectra of complex is presented in Fig 2 where as the characterization of particular protons are presented in Table 3

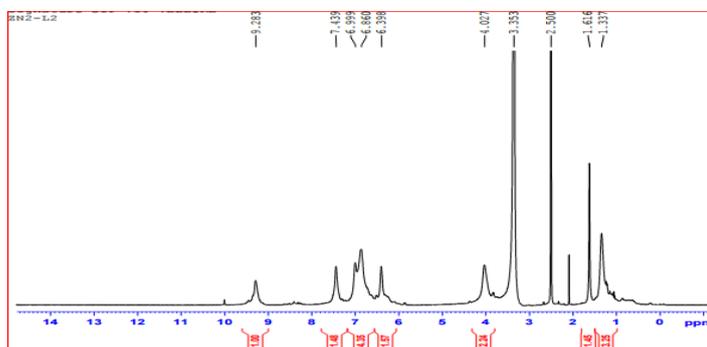


Fig:2 <sup>1</sup>HNMR Spectrum of  $\text{Ni}_2(\text{SB})_2(\text{H}_2\text{O})_2$

**Table 3**

Chemical Shift 'δ'ppm	Numbers of Protons	Multiplicity (Splitting)	Assignment
1.337	3H	S	Methyl hydrogen of Ethoxy group
4.027	2H	S	Methylene hydrogen of Ethoxy group
6.398-7.439	12H	M	Hydrogen of aromatic ring and heterocyclic pyridine
9.283	1H	S	Imine proton

The thermogram of  $\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$  complex is presented in (Fig.4). The curve reveals that there is presence of lattice as well as coordinated water molecules in the complex.

TG curve of  $\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$  complex (Fig4) shows mass loss within temperature range 50-150°C due to one lattice water molecule. An endothermic peak observed in DTA at 100 °C supports it. The second step of decomposition up to 300°C with mass loss 6.9% (calc. 7%) corresponds to elimination of two coordinated water molecules. An endothermic peak in the range 280°C was observed in the DTA curve which supports it and is attributed to the removal of two coordinated water molecules. The third step decomposition takes place up to 500°C. This decomposition suggests



removal of the organic part of the complex as four molecules of benzene ring fragments which is confirmed by an endothermic peak at 350°C and 450°C in the DTA curve.

In the fourth step sudden decomposition within temp range 500°C-700°C with weight loss of 28.5% (calc.28.9%) corresponds to decomposition of actual coordinated part of the complex above which is residue attains constant weight corresponding to metal oxide as a final product.

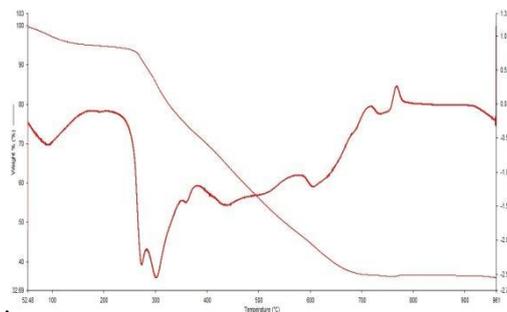


Fig:5 CuNi(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>

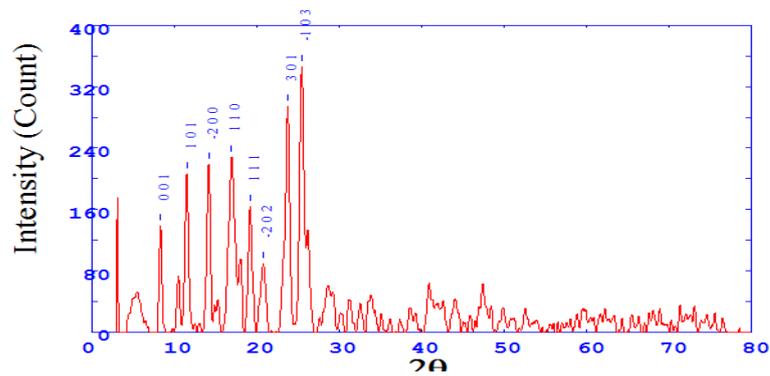
Table:4 Thermodynamic and Kinetic Parameters

Metal complex	Method	Step	Decomp. Temp.	Order of Reaction	Ea(KJ mol <sup>-1</sup> )	ΔS(KJ mol <sup>-1</sup> )	ΔG(KJ mol <sup>-1</sup> )	Zx10 <sup>-4</sup> (S <sup>-1</sup> )	Correlation Coefficient(r)
CuNi(SB) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	H-M	I	769	0.99	67.33	154.669983	78.3660297	10.337266	0.999
	C-R				64.76	152.351811	75.6254401	13.660574	0.999
	H-M	II	820	0.99	28.39	161.103398	42.0112433	5.6536042	0.999
	C-R				2912	101.550337	37.7107456	72.848973	0.999

**Powder X-ray diffraction data.**

CuCo(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> complex was used to study the X-ray powder diffraction. Diffractogram is presented in Fig. 6 The standard deviation observed is within the permissible limit. The observed density for Cu<sub>2</sub>(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> complex is 0.5gcm<sup>-3</sup> while calculated density from Z value and unit cell volume for complexes is 0.50439 gcm<sup>-3</sup> respectively. The porosity percentage calculated from the observed and calculated densities was found to 0.87035%. The crystal system was found to be tetrahedral with probable space group P. The lattice parameters reported are a = 13.48451 Å, b = 7.404824Å, c = 8.986458Å α= γ= 90° and β = 92.72° [17-18]

CuNi(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> complex was used to study the X-ray powder diffraction. Diffractogram is presented in Fig.7. The standard deviation observed is within the permissible limit. The observed density for CuNi(SB)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub> complex is 1gcm<sup>-3</sup> 0.50439 g cm<sup>-3</sup> while calculated density from Z value and unit cell volume for complexes is 0.50439 g cm<sup>-3</sup> respectively. The porosity percentage calculated from the observed and calculated densities was found to 87.31%. The crystal system was found to be tetrahedral with probable space group P. The lattice parameters reported are a = 9.854336 Å, b = 7.1256431Å, c = 7.069273Å α= γ= 90° and β = 96.18°



: Fig 6:  $\text{CuCo(SB)}_2(\text{H}_2\text{O})_2$

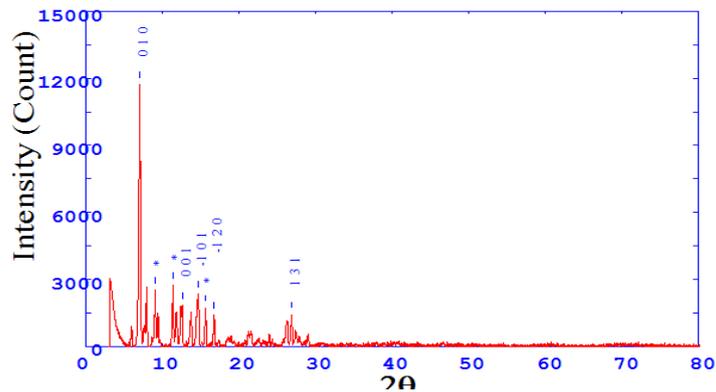


Fig.7:  $\text{CuNi(SB)}_2(\text{H}_2\text{O})_2$

**Table 5: Report for Antibacterial Testing.**

Medium-Nutrient Agar  
Method –Agar cup method

Dose of compound -1%  
cup size-10mm

Sr. No.	Test Compound	Inhibition Zone (mm)			
		<i>Escherishia coli</i>	<i>Salmonella typhi</i>	<i>Staphylococcus aureus</i>	<i>Bacillus subtilis</i>
	Penicillin	14 mm	20 mm	36 mm	28 mm
1	$\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$	16	-ve	20	-ve
2	$\text{CuCo(SB)}_2(\text{H}_2\text{O})_2$	-ve	-ve	20	20
3	$\text{CuNi(SB)}_2(\text{H}_2\text{O})_2$	-ve	20	20	20

Table 6: Report for Antifungal Testing

Test compound	Inhibit			
	<i>Aspergillus niger</i>	<i>Penicillium chrysogenum</i>	<i>Fusarium moniliforme</i>	<i>Aspergillus flavus</i>
Grisofulvin	-ve	-ve	-ve	-ve
1 $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$	-ve	+ve	-ve	-ve
2 $\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$	RG	RG	-ve	RG
3 $\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$	RG	-ve	-ve	RG

**Complex:** +ve growth = Antifungal activity absent -ve growth = Antifungal activity present  
RG = reduced growth (more than 50% reduction in growth observed)

#### Antimicrobial activity of the complexes:

The antibacterial activity of the complexes were tested against the standard microbial strains.

*Escherichia coli*, *Salmonella typhi*, *Staphylococcus aureus* and *Bacillus subtilis* by agar cup method at fixed concentration of 1% [19] and compared with known antibiotic viz *Penicillium* (Table 5). For fungicidal activity, compounds were screened in Vitro against *Aspergillus niger*, *penicillium chrysogenum*, *Fusarium moniliforme*, *Aspergillus flavus* by poison plate method with potato dextrose agar media. The complexes were tested at 1% concentration in DMSO and compared with control (Table 6)

The complexes individually show varying degrees of inhibiting effects on the growth of the bacterial species. Some complexes show activity against Gram-negative *Escherichia coli*, *Salmonella typhi* & *Bacillus subtilis*. The some complexes show activity against Gram-positive bacteria *Escherichia coli* & *Bacillus subtilis*. The metal complex  $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$  shows better activity for *Escherichia coli* however the activity of these complexes is higher than that of standard drugs. Also these complexes show better activity against *Staphylococcus aureus* but the activity is less than the standard drug. The complex  $\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$  is found to be active against *Staphylococcus aureus*, *Bacillus subtilis* bacterium. However the activity of these complexes is less than that of standard drugs. The complex  $\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$  is found to be active against *Salmonella typhi*. However the activity of these complexes is exactly equal to that of standard drugs. *Staphylococcus aureus*, *Bacillus subtilis*. However the activity of these complexes is less than that of standard drugs. Results of antifungal testing indicate that all the bimetallic complexes show moderate to high antifungal activity.

#### IV. CONCLUSION

The preparation of binuclear complexes by a novel synthetic route is strongly supported by analytical data. The formation of precursor complex as well as imines in binuclear complexes confirmed by existent and missing peaks in infrared spectra. The effective magnetic moment and electronic spectral data supported the tetrahedral environment in the metal ion. The presence of two coordinated water molecules was detected both from elemental analysis and thermo gravimetric analysis. The molecular ion peak in the mass spectra also supported the formation of binuclear complexes. Finally; the molecular mechanical method used for energy minimization corroborated the proposed structure of the complexes. The novel method to synthesize the binuclear complexes is capable of opening a new area in the preparation of complexes with a lot more variations.

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