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### Complex formation of Cr<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup> ions using 4-(2hydroxbenzylideneamino)-3-hydroxnaphtalene-1-sulfonic acid

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Schiff-base of Abstract: complexes 4 - (2 hydroxbenzylideneamino)-3-hydroxnaphtalene-1-sulfonic acid have been synthesized and investigated by elemental analysis (C.H.N.S), IR, electronic spectroscopies, molar conductivity and magnetic moment measurements. The elemental analysis data showed that all the isolated complexes have 1:1 geometry (M: L). The molar conductance values revealed that all the complexes are non-electrolytic in nature. The results of magnetic moment measurements indicate a diamagnetic nature for Ni<sup>2+</sup> complex while the complexes of Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup> are paramagnetic in nature. IR spectral data suggest the main of 4-(2-hvdroxbenzvlideneamino)-3coordination sites hydroxnaphtalene-1-sulfonic acid towards Cr3+, Co2+, Ni2+ and  $Cu^{2+}$  ions. The electronic spectral results of the ligand and its complexes show  $\pi \rightarrow \pi^*$  (phenyl ring) and  $n \rightarrow \pi^*$  (HC=N) transitions. Also the data suggest that the  $Cr^{3+}$ ,  $Co^{2+}$  and Cu<sup>2+</sup>complexes have octahedral structure while Ni<sup>2+</sup> complex has a square-planar structure. The synthesized Schiff-base and its complexes were tested against some pathogenic bacteria (Staphylococci, Proteus, Bacillus subtilis and Pseudomonas aeruginosa).

### Introduction

Schiff-bases are the condensation products of primary amines with carbonyl compounds. These compounds are known as anils, imines or azomethines (Cimerman *et al.*, 2000, Singh *et al.*, 1975, Perry *et al.*, 1988, Elmali *et al.*, 2000, Patel *et al.*, 1999, Valcarcel *et al.*, 1994, Spichiger-Keller., 1998 and Lawrence *et al.*, 1976). Schiff-bases of salicylaldehyde have been reported as plant growth regulator (Alt., 1980), antinicrobian (Hamada *et al.*, 1981), activity (Ismail, 1987). Also, Schiff-bases show some analytical applications (Palet et al., 1999). Moreover, Schiff-bases are characterized by -N=CH-(imine) group which important in elucidating the mechanism of transamination reaction in biological system (Lau et al., 1999 and Shawali et al., 1985). Schiff-bases are widely studied and used in the fields of organic synthesis and metal complexes (Toda et al., 1994 and Roland et al., 2000), for a number of their physiological reasons: and pharmacological activities; (Correa et al., 2002, Johnson et al., 1996 and Sprung., 1940)

their use in ion-selective electrodes (Naeimi et al., 2007, Ambroziak et al., 2004, Suga et al., 1998, Yang et al., 2001, Kim et al., 1999 and Sasaki et al., 1991), in the determination of heavy metal ions in environmental samples and in the extraction of metal ions (Casella et al., 1981; Waldemar et al., 1998). Some novel Schiff base ligands have been prepared through condensation of salicylaldehyde with synthetic various primary diamines under mild reaction condensations. The used aromatic diamines were synthesized in good yields starting from low-cost commercially available materials (Naeimi et al., 2015). Metal complexes of  $Cu^{2+}$ ,  $Co^{2+}$ ,  $Ni^{2+}$  and  $Zn^{2+}$  of Schiff-base derived from 5-chloro-2hydroxybenzaldehyde and 4nirobenzenesulfonamide have been prepared and characterized on the basis of physical characteristic, micro-analytical data, <sup>1</sup>H-NMR, FT-IR and UV spectrum data. The spectrum data confirm the coordination of Schiff-base with metal through imine nitrogen and oxygen The metal complexes are screened atom. against Gram-positive and Gram-negative bacteria for their biological activity. The isolated complexes show biological activity in comparison to ligand base (Khan et al., 2015).

### Experimental

### Materials

All chemicals used in this investigation were reagent of BDH or Aldrich including, 4-amino-3-hydroxy-naphthalene-1-

sulphonic acid, salicylaldehyde, EtOH, ammonium solution, ether,

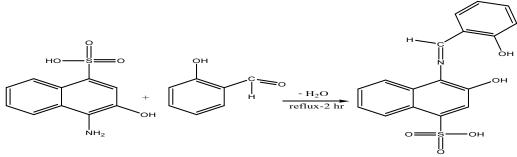
### Synthesis of Schiff base

4-(2-Hydroxbenzylideneamino)-3hydroxnaphtalene-1-sulfonic acid (H<sub>2</sub>L) was synthesized by adding (2.39 g, 0.01 mmole) of 4-amino-3-hydroxy-naphthalene-1-sulfonic acid drop wise to salicylaldehyde (1.22 g, 0.01 mmole) in 50 mL of absolute EtOH. The reaction mixture was refluxed for 3 h. The product was allowed to cool at room temperature, filtered off and recrystallized from EtOH and then dried under vacuum to get pale pink precipitate (m.p. 287 °C; yield 73%).

### Synthesis of Complexes

The Schiff-base complexes under investigation were synthesized by adding 4-(2-hydroxbenzylideneamino)-3-

hydroxnaphtalene-1-sulfonic acid (3.43 g, 0.05 mmole) in 50 mL absolute EtOH to 0.05 mmole of the salts of CrCl<sub>3</sub>.6H<sub>2</sub>O (2.66 g, 0.01 mmole), CoCl<sub>2</sub>.6H<sub>2</sub>O (2.37 g, 0.01M), NiCl<sub>2</sub>.6H<sub>2</sub>O g, (2.37)0.01M) and CuSO<sub>4</sub>.5H<sub>2</sub>O (2.49 g, 0.01M) in the same amount of the absolute EtOH or bidistilled water. The reaction mixtures were heated under reflux for 3 h. The complexes were filtered off, recrystallized from the suitable solvent and finally kept in a desiccator over silica gel.



Scheme 1

### Bacteria assay

The Schiff-base and its complexes with  $Cr^{3+}$ ,  $Co^{3+}$ ,  $Ni^{3+}$  and  $Cu^{2+}$  complexes were tested against *Pseudomonas aeruginosa* and *Bacillus subtilis* Bactetia species in a mixture of DMF and H<sub>2</sub>O. The diffusion methods were used in the antibacterial activity determination.

### **Results and discussion**

The synthesis of 4-(2hydroxbenzylideneamino)-3hydroxnaphtalene-1-sulforic acid (H<sub>2</sub>L) yields only one product (Scheme 1).

## Microanalysis and molar conductance measurements

The elemental analysis data and some physical properties of the Schiff-base and its complexes are summarized in Table 1 where the results confirm the proposed composition. The synthesized complexes were formed in 1:1 (M: L) ratio. The obtained molar conductance values of the complexes in DMF solvent lie in the range of 23.90-40.45 ohm<sup>-1</sup> cm<sup>2</sup> mol<sup>-1</sup> indicating their non-electrolytic behavior. Thus, the complexes may be formulated as [M (L)Cl<sub>n</sub>(H<sub>2</sub>O)<sub>n</sub>].nH<sub>2</sub>O where M = Cr<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup> (El-Ajaily *et al.*, 2007).

**Table (1):** Elemental analysis and some physical properties of the Schiff-base (H<sub>2</sub>L) and its complexes.

Compound	Color	M. wt.	M.P.		%( Cal	c.) (Found)		$\Lambda$ (µs)	
-			$^{0}\mathrm{C}$						BM
				C%	Н%	N%	S%		
$L(C_{17}H_{13}NO_5S)$	Pale pink	343.35	287	59.47	3.82	4.08	9.34		-
				(58.95)	(3.74)	(4.59)	(9.77)		
$[Cr(L)Cl(H_2O)_2].2H_2O$	Light green	499.99	190	40.7	3.82	2.80	6.40	29.1	3.92
				(40.62)	(4.09)	(2.25)	(5.92)		
$[Co(L)(H_2O)_3].2H_2O$	Light brown	490.02	230	41.64	4.32	2.86 (2.34)	6.54	38.3	4.78
	-			(42.31)	(4.59)		(5.99)		
[Ni(L)H <sub>2</sub> O)].3H <sub>2</sub> O	Light brown	471.01	> 250	43.25	4.06	2.97	6.79	40.5	0.0
	•			(43.72)	(4.17)	(2.46)	(6.65)		
$[Cu(L)(H_2O)_3].H_2O$	Black	476.01	180	42.81	4.02	2.94	6.72	23.9	1.8
				(43.09)	(3.22)	(3.74)	(6.30)		

# Proton nuclear magnetic resonance spectrum of ligand

The <sup>1</sup>H-NMR spectrum recorded in d<sup>6</sup> DMSO solvent on a Jeol-90 Fourier Transform (200 MHz). 4-(2-Hydroxbenzylideneamino)-3-

hydroxnaphtalene-1-sulfonic acid shows four singlet signals(figure. 1) at 11.1, 8.9, 7.9 and 6.9-7.6 ppm, downfield of TMS, assignable to the protons of OH (phenyl), OH (Sulf), H (HC=N-) and phenyl ring, respectively (Mehta *et al.*, 2007).

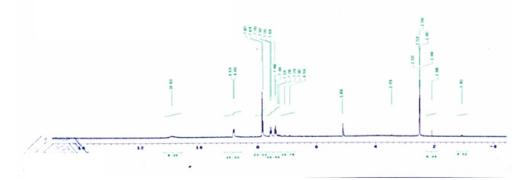


Fig.1: <sup>1</sup>H-NMR spectrum of the free ligand.

#### **Electronic spectra**

The electronic absorption spectra of the complexes were recorded using Nujol mull on a Unicam model UV2 spectrophotometer (Figs. 2- 6). The spectrum of  $Cr^{3+}$  complex exhibits three absorption bands at 23809-17241and 23809 cm<sup>-1</sup> which are due  ${}^{4}A_{2g} \rightarrow {}^{4}T_{2g}, {}^{4}A_{2g} \rightarrow$  ${}^{4}T_{1g}$  and one absorption band at 33333 cm<sup>-1</sup> due to charge-transfer. An octahedral structure was suggested for Cr<sup>3+</sup> complexes on the basis of the value of Ueff. (3.92 BM) (Elajalily et al., 2007 and Elajalily et al., 2007). The electronic absorption spectrum of the cobalt(II) complex shows three absorption bands at 31250, 22222 and 18180 cm<sup>-1</sup>, respectively, suggesting the existence of CT and d-d transitions. The band at 22222 and 18180 cm<sup>-1</sup> are assigned to the  $^{3}A_{2g} \rightarrow \ ^{3}T_{2g}$  and  $\ ^{3}A_{2g} \rightarrow \ \ ^{3}T_{1g}$  (F) transitions, respectively. The data are presented in table 2 and graphically represent in figs. 2 - 6, which

are in good agreement with the data for octahedral geometry structures as reported (Chen et al., 1995, earlier U<sub>eff.</sub> (4.78 BM) More et al., 2001 and Alan et al., 1993). The electronic absorption spectrum of the Ni(II) complex exhibits three absorptions bands, one very intestine sharp band at 34481 cm<sup>-1</sup> which attributed to ligand to metal charge-transfer transition and the other two bands laying at 22857 and 22470 cm<sup>-1</sup> which assigned to d-d transitions. No bands are observed below 10000 cm<sup>-1</sup> and hence the tetrahedral geometry can be ruled out. The complex can be considered as square-planar structure Ueff. (Zero BM) (Goel et al., 2014). The U<sub>eff</sub> Of the Cu (II) complex was found to be 1.8 BM which is very close to the spin-only value for one unpaired d electron. The bands observed in the visible region at 22222 and 18181 cm<sup>-1</sup> are attributed to  ${}^{2}B_{1g} \rightarrow {}^{2}E_{g}$  or  ${}^{2}A_{1g}$  transitions.

Table (2): IR and electronic spectral data of the ligand (H<sub>2</sub>L) and its complexes.

Ligand/ Complexes	$IR (cm^{-1})$				UV - Vis
	OH	vC=N	vM-N	vM-O	$\lambda_{\rm max}~({\rm cm}^{-1})$
$H_2L; (C_{17}H_{13}NO_5S)$	3267	1608	-	-	
[CrLCl(H <sub>2</sub> O) <sub>2</sub> ].2H <sub>2</sub> O	3315	1565	570	540	33333, 23809,17241
[CoL(H <sub>2</sub> O) <sub>3</sub> ].2H <sub>2</sub> O	3432	1565	580	550	31250, 22222, 18181
$[NiL(H_2O)].3H_2O$	3432	1527	580	445	34481, 22857, 22470
$[CuL(H_2O)_3].H_2O$	3445	1565	505	515	31250, 22222, 18181

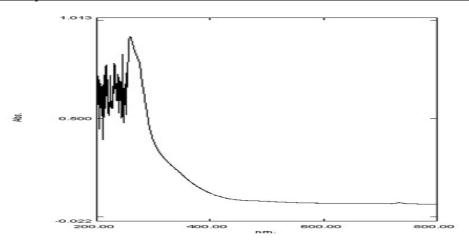
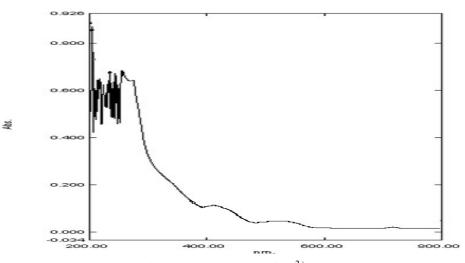
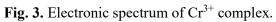
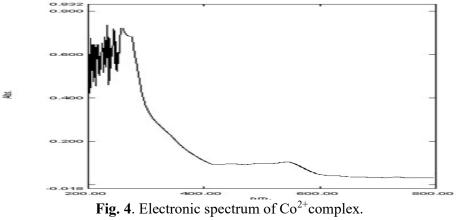
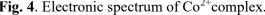


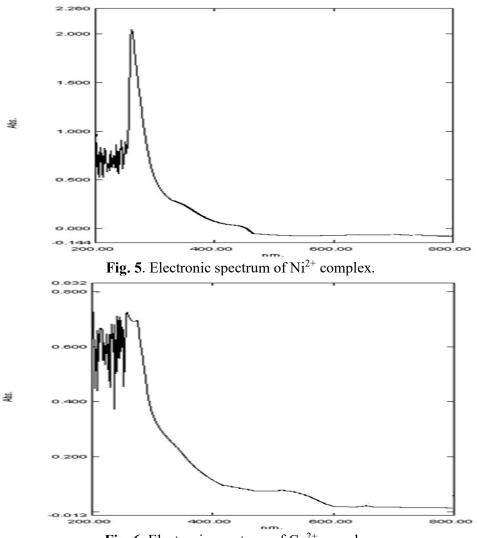
Fig. 2. UV spectrum of the free ligand (H<sub>2</sub>L).











**Fig. 6**. Electronic spectrum of  $Cu^{2+}$  complex.

### **IR** spectra

The IR spectra of the ligand and its complexes with Cr<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup> were recorded in the solid state in the rang 400-4000 cm<sup>-1</sup> using KBr disc on a Perkin-Elmer 1430 ratio recording infrared spectrophotometer (Figs. 7-10). The IR spectral data are present in Table 2. The results show very strong band at 1608 cm<sup>-1</sup> for (C = N) group of ligand the lowering of v(C=N) group by  $40 \text{ cm}^{-1}$  in all complexes indicates the participation of nitrogen atom during chelates. No bands in region 3400-3200 cm<sup>-1</sup> in the metal complexes indicate the absence of coordinated H2O molecule. The band in the range 1009-982 cm<sup>-1</sup> is attributed to Cr-O. The bands around 580-515 cm<sup>-1</sup> are assigned to v(M-N) and v(M-O)vibrations for Cr<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup>

complexes, respectively. The IR spectrum of the ligand shows two intense absorption bands at 3257cm<sup>-1</sup> and 1608 cm<sup>-1</sup> assigned to the free v(OH) (Rpudi *et al.*, 1989) and v(C=N)vibrations, respectively. In case of complexes with  $Cr^{3+}$ ,  $Co^{2+}$ ,  $Ni^{2+}$  and  $Cu^{2+}$  the absorption bands of water have been shifted to 3315 cm<sup>-1</sup>,  $3432 \text{ cm}^{-1}$ ,  $3432 \text{ cm}^{-1}$  and  $3445 \text{ cm}^{-1}$ selectively, also another absorption band is observed at 1608 cm<sup>-1</sup> for v(C=N) of the free ligand which attribute to v(C=N) stretching vibration and were shifted to 1565cm<sup>-1</sup>, 1565 cm<sup>-1</sup>, 1527 cm<sup>-1</sup> and 1565 cm<sup>-1</sup> for Cr<sup>3+</sup>, Co<sup>2+</sup>, Ni<sup>2+</sup> and Cu<sup>2+</sup> complexes, respectively, the bands undergo Rigalne shift on complexes. (Agarwal et al., 1991, Juneja et al., 1988, Vivekanand, 2007). All these observations confirm that the ligand (H<sub>2</sub>L) behaves as dibasic tridentate ligand, coordinating through

nitrogen atom of HC=N and both oxygen atoms of the deprotonated (OH) phenyl group

as shown in structures 1-5.

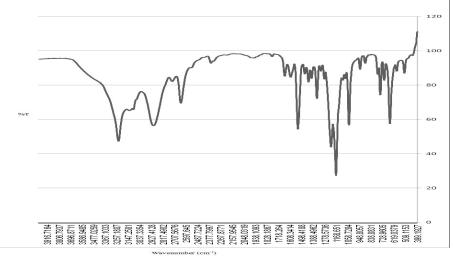
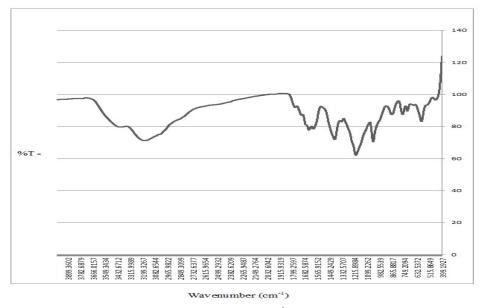
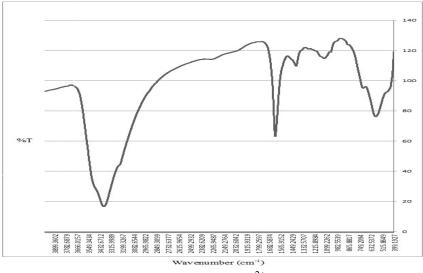
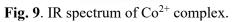


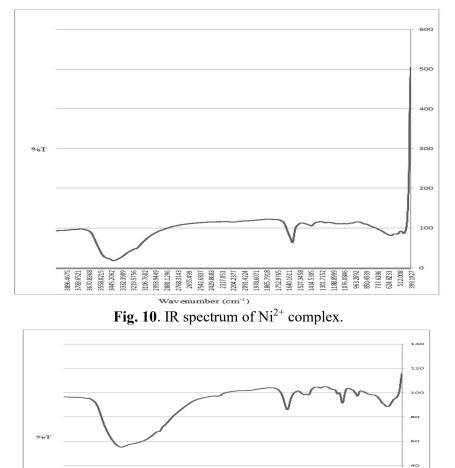
Fig. 7. IR spectrum of the free ligand (H<sub>2</sub>L) in KBr.



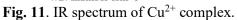
**Fig. 8**. IR spectrum of  $Cr^{3+}$  complex.











### Antibacterial activity

The Schiff-base and its complexes showed inhibitory activity against all used

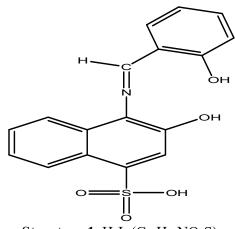
bacteria species (*Pseudomonas aeruginosa* and *Bacillus subtilis* bacteria) for the free Schiffbase 17-16 mm and 16-10 mm for complexes, the antibacterial results (mm) are presented in Table 3.

Table (3): Antibacterial activity results (mm) of Schiff-base and its comp
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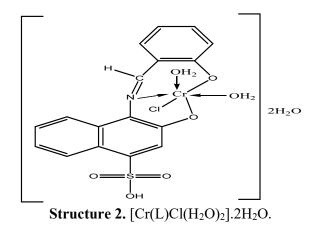
	Bacteria			
L and its complexes	Pseudomonas Aeruginosa	Bacillus Subtilis		
$H_2L; (C_{17}H_{13}NO_5S)$	17	16		
$[CrL(Cl)(H_2O)_2].2H_2O$	15	11		
$[CoL(H_2O)_3].2H_2O$	14	10		
[NiLH <sub>2</sub> O)].3H <sub>2</sub> O	12	11		
$[CuL(H_2O)_3].H_2O$	16	12		

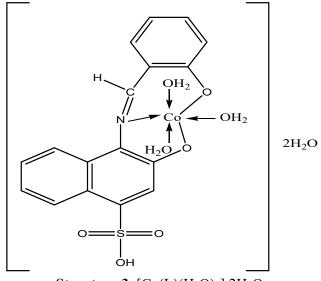
On the basis of the elemental composition, electronic and IR spectral studies the following

structures (1-5) are proposed for the ligand synthesized complexes.

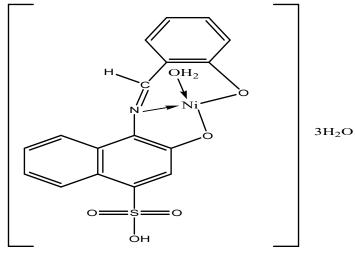


Structure 1.  $H_2L$  ( $C_{17}H_{13}NO_5S$ ).

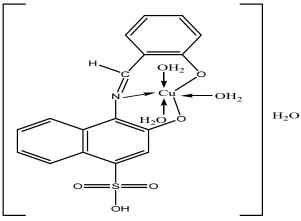




**Structure 3.** [Co(L)(H<sub>2</sub>O)<sub>3</sub>].2H<sub>2</sub>O.



Structure 4. [Ni(L)H<sub>2</sub>O)].3H<sub>2</sub>O.



**Structure 5**. [Cu(L)(H<sub>2</sub>O)<sub>3</sub>].H<sub>2</sub>O.

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### تحضير متراكبات Cr(III), Co(II), Ni(II), Cu(II) مع مرتبط 4-(2- هيدروكسي بنزيل ايدين امينو)-3- هيدروكس نفثالين -1- حمض الكبريتيك.

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تم تحضير مرتبط قاعدة شيف من تكثيف 4- امينو -3- هيدروكسي -1- حمض الكبريتيك مع السلسلدهيد وتمت دراسته بواسطة التقنيات مثل التحليل العنصري والأشعة فوق بنفسجية والأشعة تحت الحمراء والرنين النووي المغناطيسي ومطياف الكتلة, وحضر منها متراكبات لقواعد شيف بتكثيفها مع أيونات Co(II), Ni(II), Cu(II) الثنائي وأيون (Cr(III) وتم تحديد الأشكال الفراغية للمتراكبات بواسطة الطرق الطيفية والمغناطيسية, وإقترحت جميع المتراكبات ثمانية السطوح, كما أجريت لها تطبيقات على بعض أنواع من البكتيريا Pseudomonas aeruginosa and Bacillus subtilis bactetia .