



Complex formation of Cr^{3+} , Co^{2+} , Ni^{2+} and Cu^{2+} ions using 4-(2-hydroxybenzylideneamino)-3-hydroxynaphtalene-1-sulfonic acid

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Received 24 June 2015; accepted 1 September 2015

Keywords

4-(2-hydroxybenzylideneamino)-3-hydroxynaphtalene-1-sulfonic acid; Complexes.

Abstract: Schiff-base complexes of 4-(2-hydroxybenzylideneamino)-3-hydroxynaphtalene-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^{2+} complex while the complexes of Co^{2+} , Ni^{2+} and Cu^{2+} are paramagnetic in nature. IR spectral data suggest the main coordination sites of 4-(2-hydroxybenzylideneamino)-3-hydroxynaphtalene-1-sulfonic acid towards Cr^{3+} , Co^{2+} , Ni^{2+} 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^{2+} complexes have octahedral structure while Ni^{2+} 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), antimicrobial (Hamada *et al.*, 1981), activity (Ismail, 1987).

Also, Schiff-bases show some analytical applications (Palet *et al.*, 1999). Moreover, Schiff-bases are characterized by $-\text{N}=\text{CH}-$ (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 reasons: their physiological 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-2-hydroxybenzaldehyde and 4-nirobenzenesulfonamide have been prepared and characterized on the basis of physical characteristic, micro-analytical data, $^1\text{H-NMR}$, FT-IR and UV spectrum data. The spectrum data confirm the coordination of Schiff-base with metal through imine nitrogen and oxygen atom. The metal complexes are screened 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-

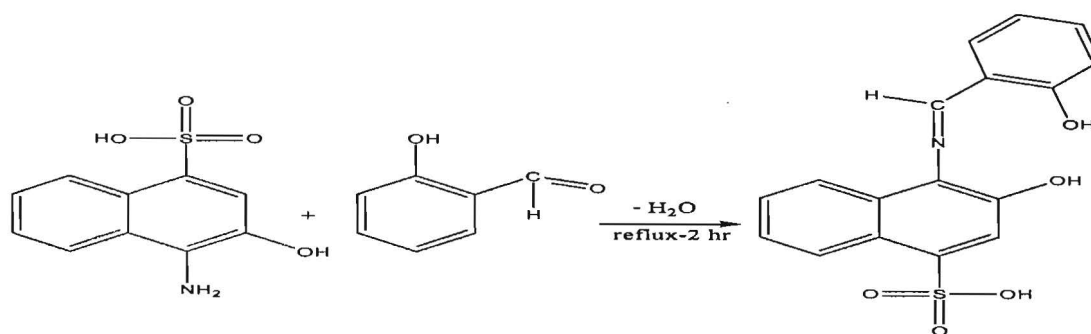
sulphonic acid, salicylaldehyde, EtOH, ammonium solution, ether,

Synthesis of Schiff base

4-(2-Hydroxybenzylideneamino)-3-hydroxynaphthalene-1-sulfonic acid (H_2L) 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-hydroxybenzylideneamino)-3-hydroxynaphthalene-1-sulfonic acid (3.43 g, 0.05 mmole) in 50 mL absolute EtOH to 0.05 mmole of the salts of $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ (2.66 g, 0.01 mmole), $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (2.37 g, 0.01M), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (2.37 g, 0.01M) and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (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_2O . The diffusion methods were used in the antibacterial activity determination.

Results and discussion

The synthesis of 4-(2-hydroxybenzylideneamino)-3-hydroxynaphtalene-1-sulfonic acid (H_2L) 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 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ indicating their non-electrolytic behavior. Thus, the complexes may be formulated as $[\text{M}(\text{L})\text{Cl}_n(\text{H}_2\text{O})_n] \cdot n\text{H}_2\text{O}$ where $\text{M} = \text{Cr}^{3+}$, Co^{2+} , Ni^{2+} and Cu^{2+} (El-Ajaily *et al.*, 2007).

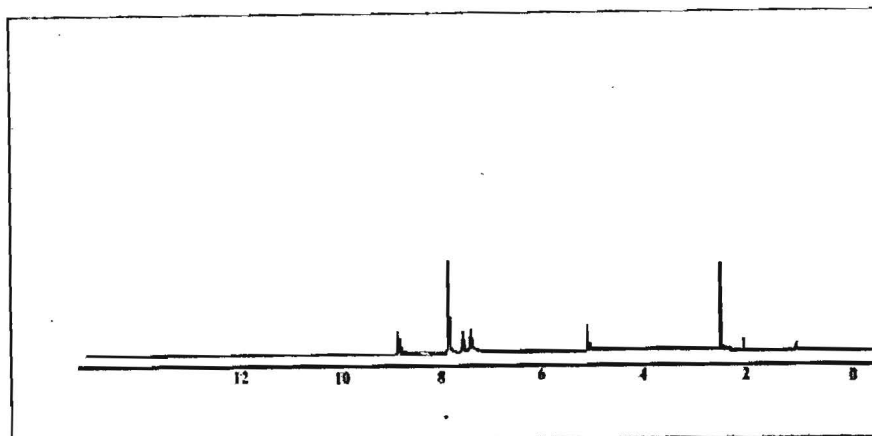
Table (1): Elemental analysis and some physical properties of the Schiff-base (H_2L) and its complexes.

Compound	Color	M. wt.	M.P. °C	%(Calc.) (Found)				Λ (μs)	BM
				C%	H%	N%	S%		
L ($\text{C}_{17}\text{H}_{13}\text{NO}_5\text{S}$)	Pale pink	343.35	187	59.47 (58.95)	3.82 (3.74)	4.08 (4.59)	9.34 (9.77)	-	-
$[\text{Cr}(\text{L})\text{Cl}(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$	Light green	499.99	190	40.7 (40.62)	3.82 (4.09)	2.80 (2.25)	6.40 (5.92)	29.1	3.92
$[\text{Co}(\text{L})(\text{H}_2\text{O})_3] \cdot 2\text{H}_2\text{O}$	Light brown	490.02	230	41.64 (42.31)	4.32 (4.59)	2.86 (2.34)	6.54 (5.99)	38.3	4.78
$[\text{Ni}(\text{L})(\text{H}_2\text{O})] \cdot 3\text{H}_2\text{O}$	Light brown	471.01	> 250	43.25 (43.72)	4.06 (4.17)	2.97 (2.46)	6.79 (6.65)	40.5	0.0
$[\text{Cu}(\text{L})(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$	Black	476.01	180	42.81 (43.09)	4.02 (3.22)	2.94 (3.74)	6.72 (6.30)	23.9	1.8

Proton nuclear magnetic resonance spectrum of ligand

The ^1H -NMR spectrum recorded in d^6 DMSO solvent on a Jeol-90 Fourier Transform (200 MHz). 4-(2-Hydroxybenzylideneamino)-3-

hydroxynaphtalene-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: ^1H -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 23809-17241 and 23809 cm^{-1} which are due $^4\text{A}_{2g} \rightarrow ^4\text{T}_{2g}$, $^4\text{A}_{2g} \rightarrow ^4\text{T}_{1g}$ and one absorption band at 33333 cm^{-1} due to charge-transfer. An octahedral structure was suggested for Cr^{3+} complexes on the basis of the value of U_{eff} . (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^{-1} , respectively, suggesting the existence of CT and d-d transitions. The band at 22222 and 18180 cm^{-1} are assigned to the $^3\text{A}_{2g} \rightarrow ^3\text{T}_{2g}$ and $^3\text{A}_{2g} \rightarrow ^3\text{T}_{1g}$ (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 earlier U_{eff} . (4.78 BM) (Chen *et al.*, 1995, 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^{-1} which attributed to ligand to metal charge-transfer transition and the other two bands laying at 22857 and 22470 cm^{-1} which assigned to d-d transitions. No bands are observed below 10000 cm^{-1} and hence the tetrahedral geometry can be ruled out. The complex can be considered as square-planar structure U_{eff} . (Zero BM) (Goel *et al.*, 2014). The U_{eff} . 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^{-1} are attributed to $^2\text{B}_{1g} \rightarrow ^2\text{E}_g$ or $^2\text{A}_{1g}$ transitions.

Table (2): IR and electronic spectral data of the ligand (H_2L) and its complexes.

Ligand/ Complexes	IR (cm^{-1})				UV - Vis λ_{max} (cm^{-1})
	OH	$\nu\text{C}=\text{N}$	$\nu\text{M}-\text{N}$	$\nu\text{M}-\text{O}$	
$\text{H}_2\text{L}; (\text{C}_{17}\text{H}_{13}\text{NO}_5\text{S})$	3267	1608	-	-	
$[\text{CrLCl}(\text{H}_2\text{O})_2].2\text{H}_2\text{O}$	3315	1565	570	540	33333, 23809, 17241
$[\text{CoL}(\text{H}_2\text{O})_3].2\text{H}_2\text{O}$	3432	1565	580	550	31250, 22222, 18181
$[\text{NiL}(\text{H}_2\text{O})_3].3\text{H}_2\text{O}$	3432	1527	580	445	34481, 22857, 22470
$[\text{CuL}(\text{H}_2\text{O})_3].\text{H}_2\text{O}$	3445	1565	505	515	31250, 22222, 18181

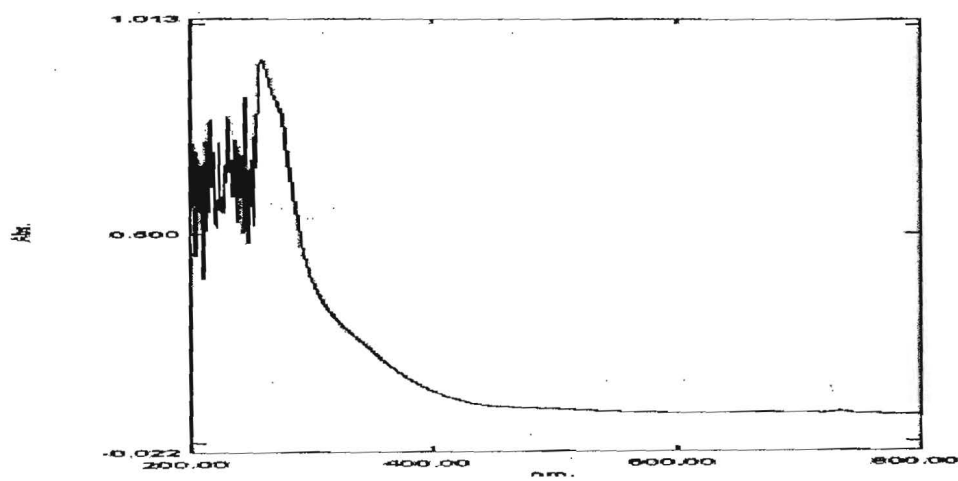


Fig. 2. UV spectrum of the free ligand (H_2L).

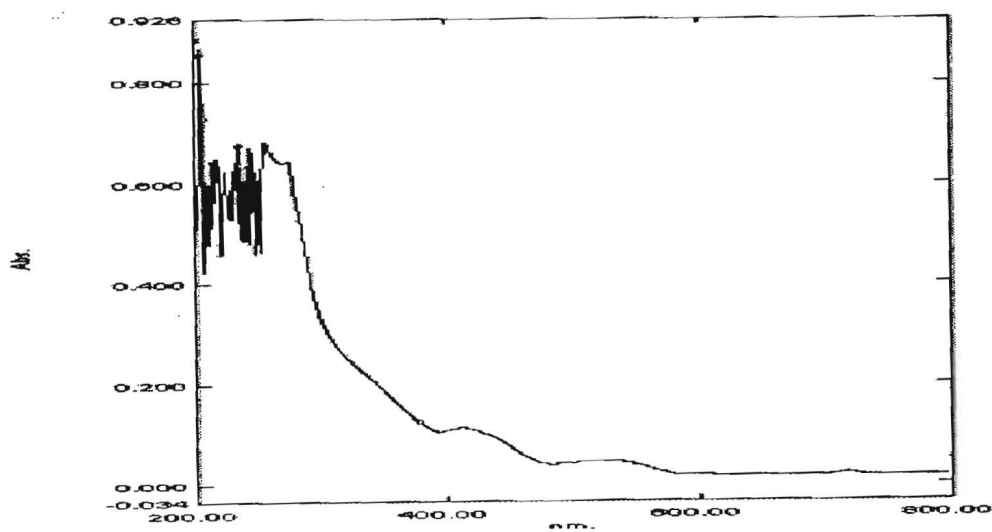
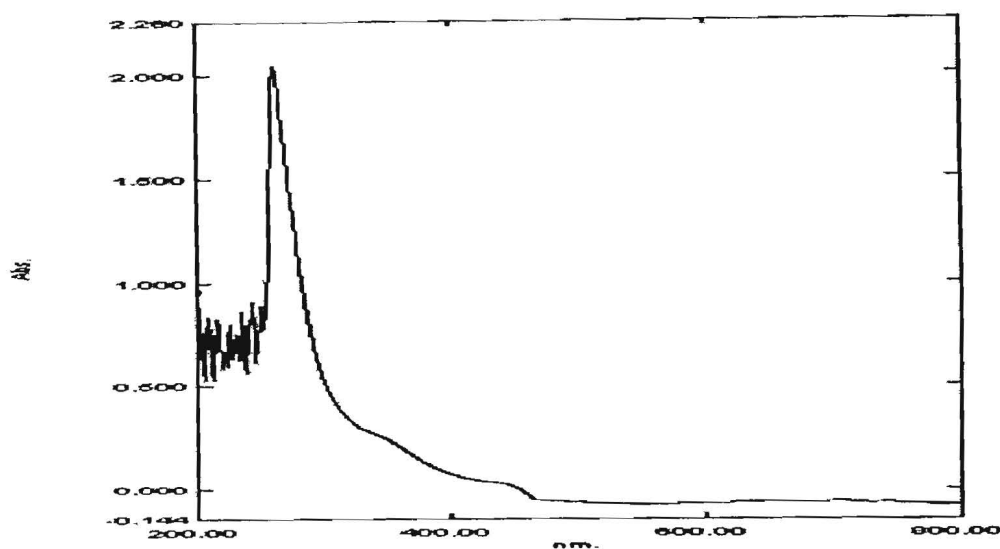
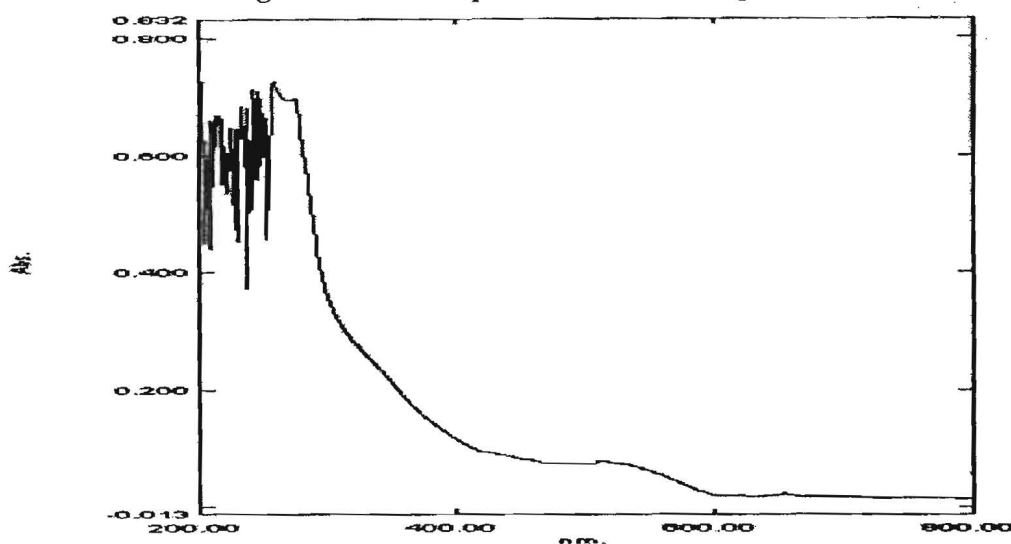


Fig. 3. Electronic spectrum of Cr^{3+} complex.



Fig. 4. Electronic spectrum of Co^{2+} complex.

Fig. 5. Electronic spectrum of Ni²⁺ complex.Fig. 6. Electronic spectrum of Cu²⁺ complex.

IR spectra

The IR spectra of the ligand and its complexes with Cr³⁺, Co²⁺, Ni²⁺ and Cu²⁺ were recorded in the solid state in the range 400-4000 cm⁻¹ 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⁻¹ for (C = N) group of ligand the lowering of $\nu(\text{C}=\text{N})$ group by 40 cm⁻¹ in all complexes indicates the participation of nitrogen atom during chelates. No bands in region 3400-3200 cm⁻¹ in the metal complexes indicate the absence of coordinated H₂O molecule. The band in the range 1009-982 cm⁻¹ is attributed to Cr-O. The bands around 580-515 cm⁻¹ are assigned to $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$ vibrations for Cr³⁺, Co²⁺, Ni²⁺ and Cu²⁺

complexes, respectively. The IR spectrum of the ligand shows two intense absorption bands at 3257 cm⁻¹ and 1608 cm⁻¹ assigned to the free $\nu(\text{OH})$ (Rpudi *et al.*, 1989) and $\nu(\text{C}=\text{N})$ vibrations, respectively. In case of complexes with Cr³⁺, Co²⁺, Ni²⁺ and Cu²⁺ the absorption bands of water have been shifted to 3315 cm⁻¹, 3432 cm⁻¹, 3432 cm⁻¹ and 3445 cm⁻¹ selectively, also another absorption band is observed at 1608 cm⁻¹ for $\nu(\text{C}=\text{N})$ of the free ligand which attribute to $\nu(\text{C}=\text{N})$ stretching vibration and were shifted to 1565 cm⁻¹, 1565 cm⁻¹, 1527 cm⁻¹ and 1565 cm⁻¹ for Cr³⁺, Co²⁺, Ni²⁺ and Cu²⁺ 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 (H₂L) 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.

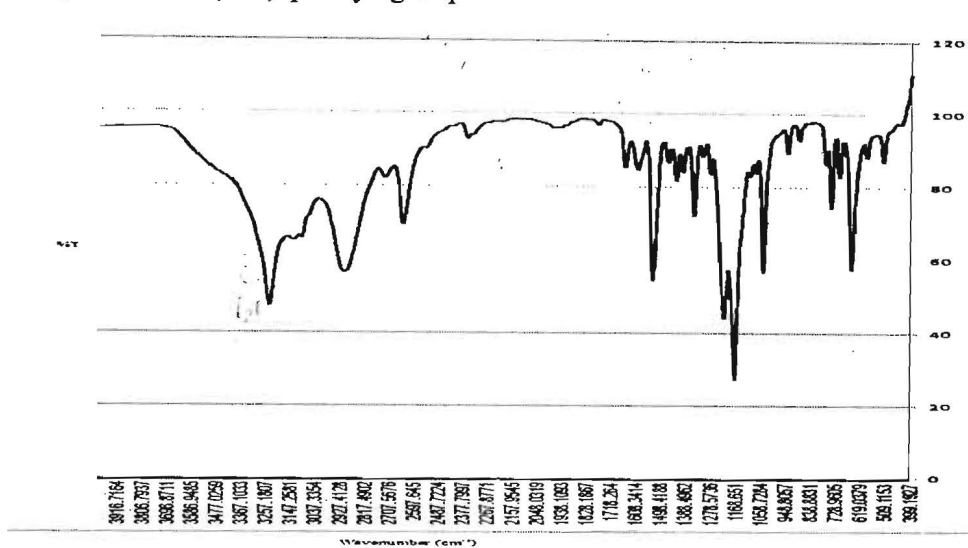


Fig. 7. IR spectrum of the free ligand (H_2L) in KBr.

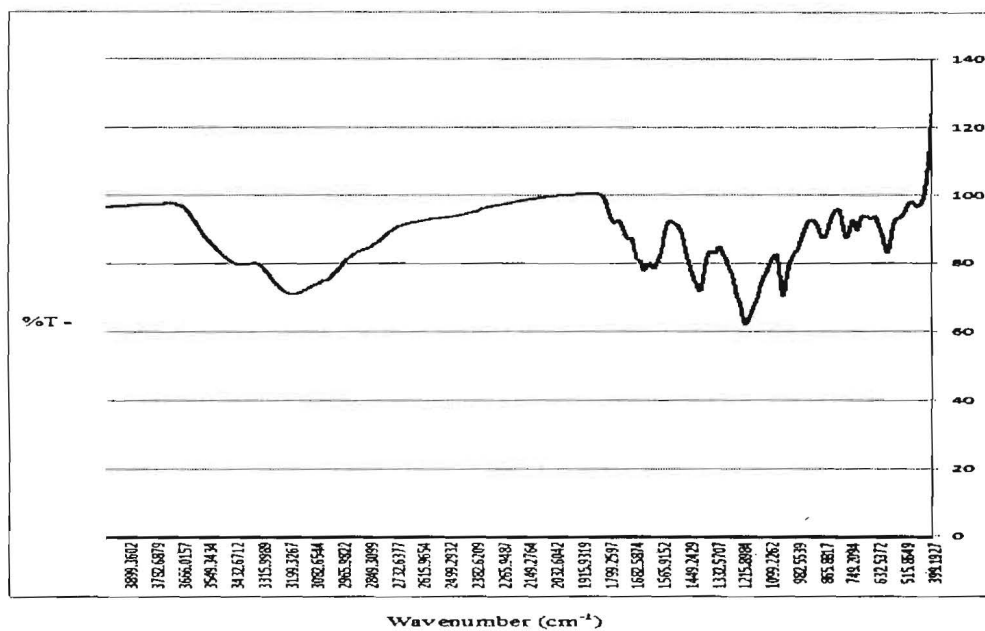


Fig. 8. IR spectrum of Cr^{3+} complex.

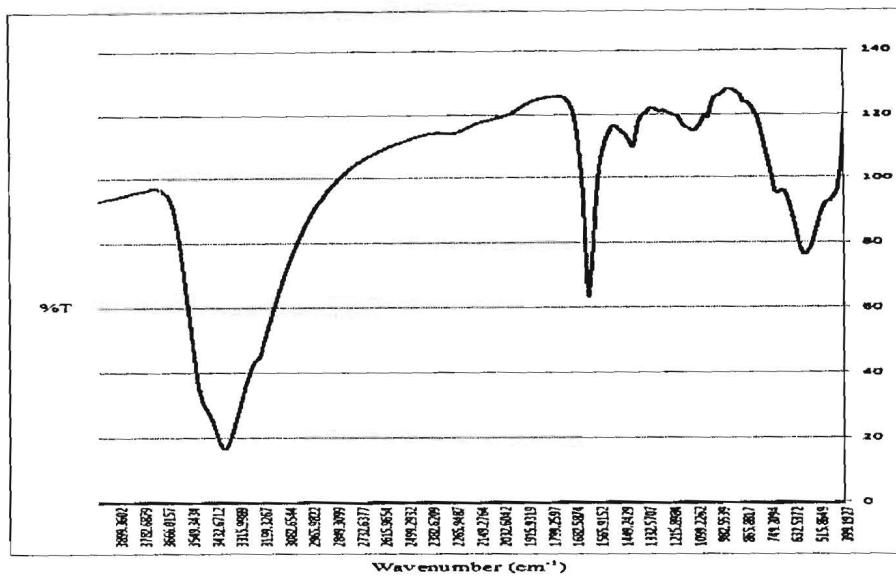


Fig. 9. IR spectrum of Co²⁺ complex.

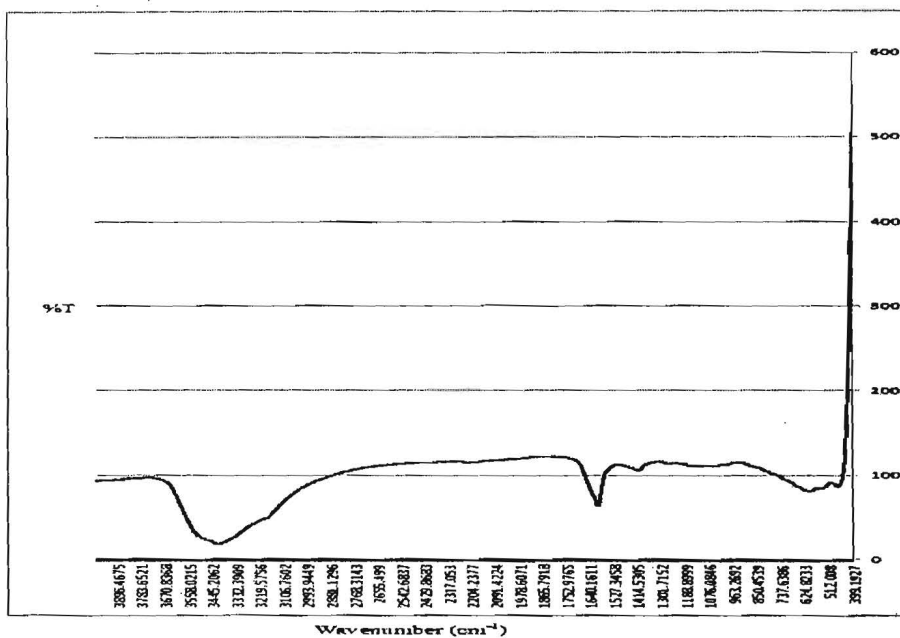


Fig. 10. IR spectrum of Ni²⁺ complex.

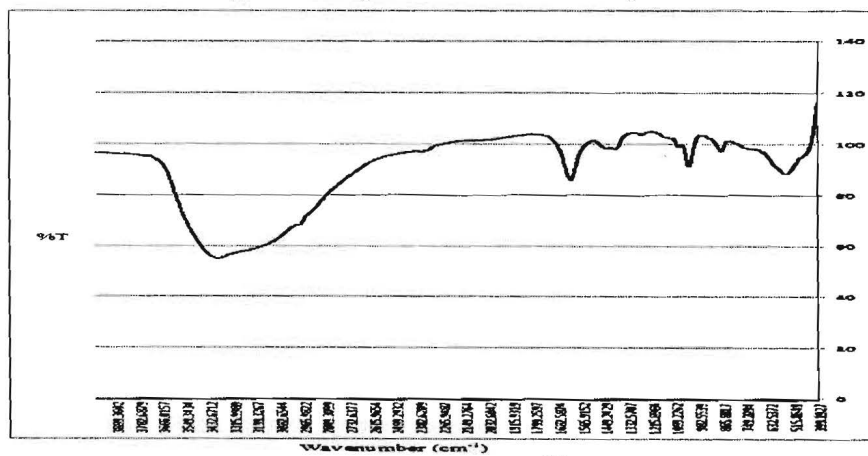


Fig. 11. IR spectrum of Cu²⁺ complex.

Antibacterial activity

The Schiff-base and its complexes showed inhibitory activity against all used bacteria species (*Pseudomonas aeruginosa* and

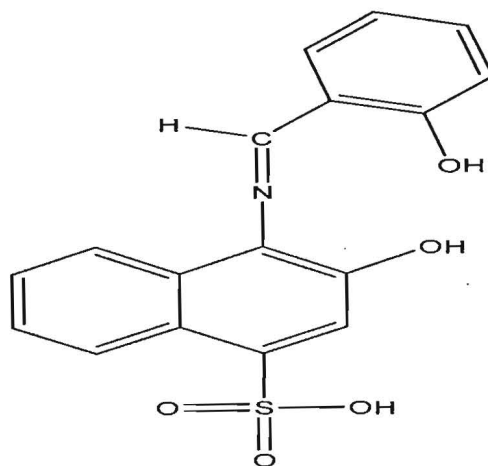
Bacillus subtilis bacteria) for the free Schiff-base 17-16 mm and 16-10 mm for complexes, the antibacterial results (mm) are presented in Table 3.

Table (3): Antibacterial activity results (mm) of Schiff-base and its complexes.

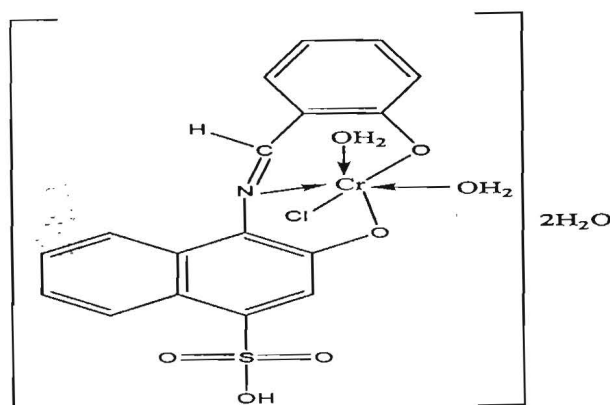
L and its complexes	Bacteria	
	<i>Pseudomonas Aeruginosa</i>	<i>Bacillus Subtilis</i>
H ₂ L; (C ₁₇ H ₁₃ NO ₅ S)	17	16
[CrL(Cl)(H ₂ O) ₂].2H ₂ O	15	11
[CoL(H ₂ O) ₃].2H ₂ O	14	10
[NiLH ₂ O].3H ₂ O	12	11
[CuL(H ₂ O) ₃].H ₂ O	16	12

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

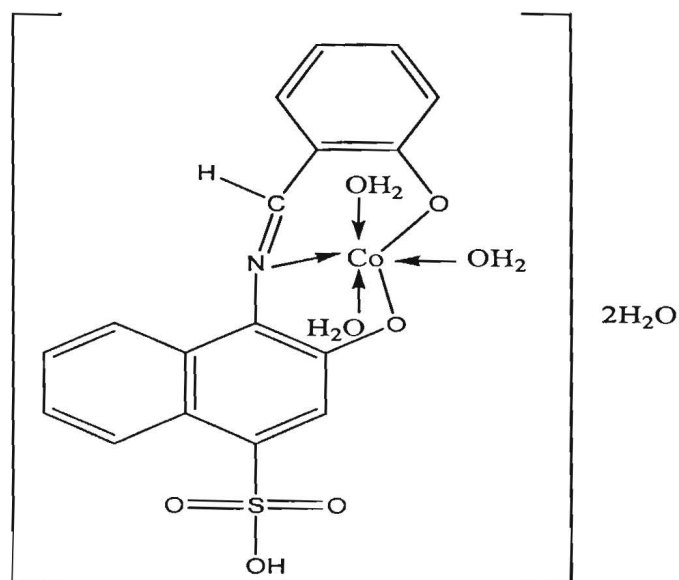
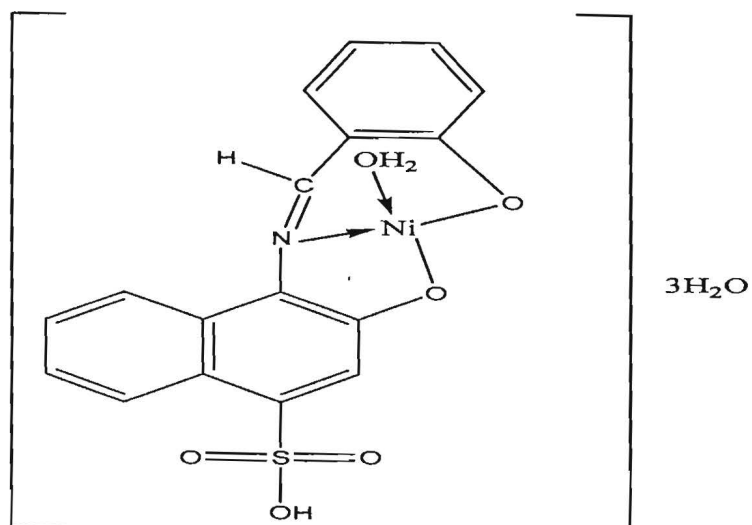
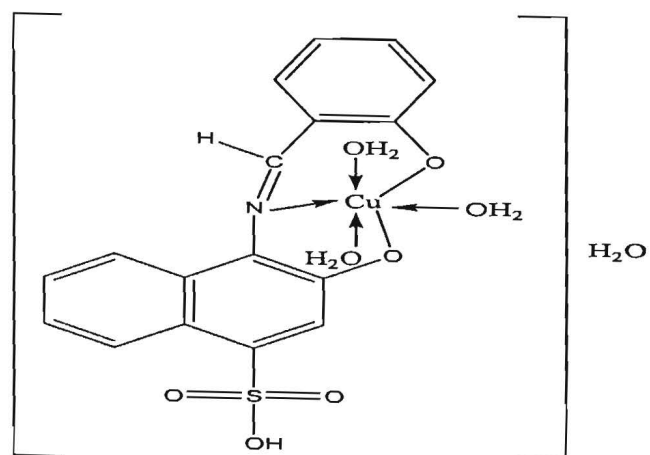
structures (1-5) are proposed for the ligand synthesized complexes.



Structure 1. H₂L (C₁₇H₁₃NO₅S).



Structure 2. [Cr(L)Cl(H₂O)₂].2H₂O.

**Structure 3.** [Co(L)(H₂O)₃].2H₂O.**Structure 4.** [Ni(L)H₂O].3H₂O.**Structure 5.** [Cu(L)(H₂O)₃].H₂O.

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تحضير مترابطات Cr(III) ، Co(II), Ni(II), Cu(II) مع مرتبط ٤-٢) - هيدروكسي بنزويل ايدين امينو) -٣- هيدروكس نفتالين -١- حمض الكبريتيك.

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تم تحضير مرتبط قاعدة شيف من تكثيف ٤- امينو -٣- هيدروكسي -١- حمض الكبريتيك مع السلسلهيد وتمت دراسته بواسطة التقنيات مثل التحليل العنصري والأشعة فوق بنفسجية والأشعة تحت الحمراء والرنين النووي المغناطيسي ومطياف الكتلة، وحضر منها مترابطات لقواعد شيف بتكثيفها مع أيونات Co(II), Ni(II), Cu(II) الثنائي وأيون Cr(III) وتم تحديد الأشكال الفراغية للمترابطات بواسطة الطرق الطيفية والمغناطيسية، وإقترحت جميع المترابطات ثمانية السطوح، كما أجريت لها تطبيقات على بعض أنواع من البكتيريا *Pseudomonas aeruginosa* and *Bacillus subtilis* .