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Original Article

Mixed ligand chelates using Schiff base and 2-aminobenzoic acid as ligands

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Abstract

The ligands under investigation were used to form five mixed ligand chelates with some transition metal ions. The ligands and their chelates were offered to several analytical and spectroscopic tools. Based on the obtained results; an octahedral structure was proposed for all the chelates. Also, the antibacterial activity was tested on two types of bacteria; Staphylococcus aureus and E.coli species.

Keywords: Schiff base, mixed ligand complexes, physiochemical characterization

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Introduction

Schiff base ligands and their transition metal complexes have covered a large field in chemistry that is being noticed. Schiff base ligands are viewed as special ligands since they are easily prepared by condensation reactions between aldehyde derivatives and amines [1]. Schiff bases are the compounds containing the azomethine group (-HC=N-). They are condensation products of ketones (or) aldehydes (aldehydes and ketones) with primary amines and were first reported by Hugo Schiff in 1864 [2]. Metal complexes of Co(II), Cu(II) and Zn(II) ions of Schiff base have played a central role in the development of coordination chemistry [3]. Mixed ligand complexes of Cu(II), Co(II), Ni(II), Mn(II), and Cd(II) with N, N'-1,4-phenylene (2-hyboxynaphthalidenimine) (H2L) as primary ligand and 4-aminoantipyrine (4-AAp) as secondary ligand have been prepared. The newly formed complexes were characterized using elemental analysis, magnetic susceptibility, molar conductance, 1H

NMR, UV–Vis., FTIR, X-ray diffraction, mass, thermal analysis and ESR techniques. The elemental analysis and data reveal the formation of [2:1:2] [M: H2L: 4-AAP] complexes. The mode of bonding between Cu (II), Ni (II), Co (Il), Mn (II) and Cd (II) and ligands has been studied by IR spectrophotometry. Electronic absorption spectra and magnetic moment measurements of the mixed-ligand complexes suggest a distorted octahedral geometry around the central metal ion and the anions X− are in the axial positions, for all compounds [4]. Novel three mixed ligand complexes derived from the interaction of Cr (III), Fe (III) and La (III) ions with a Schiff base resulted from the condensation process of 2-aminophenol and 2-hydroxyacetophenone (HL1) as a primary ligand (HL1) and anthranilic acid (L2) as a secondary ligand. The formed mixed ligand complexes were synthesized and characterized using elemental analysis, FTIR, 1HNMR, UV–visible and mass spectroscopy as well as molar conductance and magnetic measurements. The obtained results revealed that the mixed ligand complexes were formed in a 1:1:1[L1ML2] ratio and were non-electrolytic [5].

The present study aims to synthesize five mixed ligand chelates of Mn (II), Co (II), Fe(III), Ni (II), and Cr (III) ions with two ligands: The first ligand is a Schiff base that is derived from the condensation of 2-hydroxyacetophenone with 2,4-dinitrophenyl hydrazine, which serves as the Primary ligand, whereas the secondary ligand is with 2-aminobenzoic acid. Moreover, their characterization and antibacterial activity were also studied.

Material and Methods

Chemicals and reagents

All chemicals and solvents used in this study were of analar Grade (BDH, Aldrich). They include 2,4-dinitrophnylhydrazine, 2-aminobenzoic acid, 2-hydroxyaceto-phenone, and some metal salts; Cr Cl3.6H2O, Fe(NO3)3.6H2O, CoCl2.6H2O, NiCl2.6H2O, MnCl2.4H2O, DMF, glacial acetic acid, absolute ethanol and ether. These solvents were either spectroscopically pure.

Preparation of the Schiff base

The Schiff base was prepared by adding (6.808g, 0.05 mmole) of 2-hydro-xyacetophenone dropwise to 2,4-dinitrophnylhydrazine (9.907 g, 0.05 mmol) in 50 mL of absolute ethanol. The reaction mixture was refluxed for 3 hours. Then the product was cooled at room temperature, filtered off and recrystallized from ethanol and dried under vacuum to get a yellow precipitate. The Schiff base formation is shown in Fi. 1.

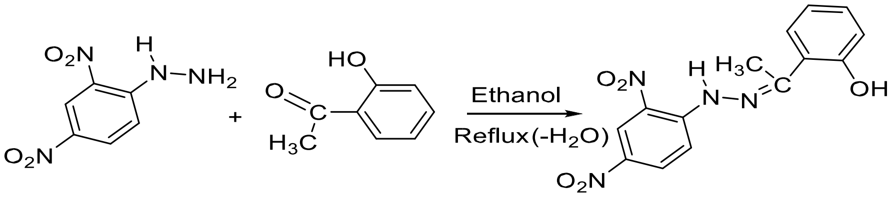


Fig. 1: Formation of Schiff base

Synthesis mixed ligand chelates

The mixed ligand chelates were Synthesized from 30 mL absolute ethanol solution of the Schiff base (15.81g, 0.05mmol), 30mL absolute ethanol solution (13.322g, 0.1mmol) of CrCl3.6H2O, (9.896 g, 0.05 mmol) MnCl2.4H2O, Fe(NO3)3.6H2O (17.50g,0.05mmol), CoCl2.6H2O (11.90g, 0.05 mmol) and NiCl2. 6H2O (11.885g, 0.05 mmol) was added after 10 minutes 30 mL of the same solvent of 2-aminobenzoic acid (4.898 mL, 0.05 mmol) was added dropwise as a secondary ligand (L2) and the resulting mixtures were refluxed for about 3 hours. The obtained precipitates were filtered, washed with ethanol and dried under vacuum on anhydrous CaCl2.

Antibacterial assay

The antibacterial activity of the mixed ligand chelates was tested against two kinds of bacteris species; Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli). These species were cultured in Muller Hinton Agar Media and the disk diffusion method was employed to assess the antibacterial activity of the mixed ligand chelates. Sterile paper disks were impregnated with concentrations of the chelates (100mg/ml) and the disks were then placed on agar plates pre-inoculated with the respective bacterial cultures suspension of McFarland(0.5). The plates were incubated at 370C for 24 hrs to allow for bacterial growth and formation of inhibition zones. The zone diameters of inhibition around each disk were measured in mm[6].

Results and Discussion

CHN elemental analyses and molar conductivity

The physical properties and elemental analysis data of the Schiff-base and mixed ligand complexes are summarized in Table 1, where, the results confirm the proposed composition. The synthesized mixed ligand complexes were formed in 1:1:2 or 1:1:1 (L1:M:L2) ratio. The obtained molar conductance values of the complexes in DMF solvent lie in the range of 09-18 ohm-1 cm2 mol-1 indicating their non-electrolytic behavior [7].

Table (1): Some physical properties and elemental analyses of Schiff base and mixed ligand chelates

Compound/ mixed ligand chelates	Color	M.wt.	M.P.°C	%Calc. (Found)				Λ_m Ω-1 cm^{2}.mol^{-1}	μs (BM)
				C%		H%	N%		
Schiff base (HL1)	Orange	316.27	210	53.17 **(52.09)**		3.82 **(3.62)**	17.72 **(17.11)**	-	-

[Cr (L1) (L2)$_2$]	Dark Orange	639.52	216-217	52.59 **(52.69)**		3.63 **(4.07)**	13.14 **(13.04)**	19	3.77
Mn(L1)(L2)(H$_2$O)$_2$]	Light brown	542.36	208-210	46.51 **(46.58)**		3.90 **(4.09)**	12.91 **(13.65)**	12	5.67
[Fe(L1)(L2) (NO$_3$) (H$_2$O)]	Blackish brown	543.27	210-213	46.43 **(47.12)**		3.90 **(4.48)**	12.89 **(13.02)**	10	4.90
[Co(L1)(L2) (H$_2$O)$_2$]	Brown	546.36	210-213	46.17 **(46.56)**		3.87 (4.12)	12.82 **(12.95)**	15	3.85
[Ni(L1)(L2) (H$_2$O)$_2$]	Dark brown	546.12	215-218	46.19 **(47.09)**		3.88 **(3.62)**	10.75 **(10.11)**	13	2.82

Mass spectrum of Schiff base

The mass spectral data of the Schiff base ligand is demonstrated in Figure 2. Molecular ions showed peaks that were in good agreement with the expected

Values [8]. The mass spectrum gives a base peak at 316 m/z.

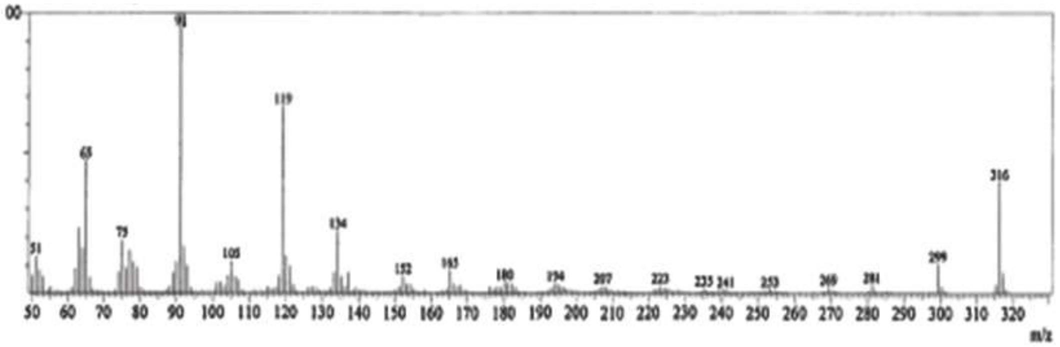


Fig. 2: Mass spectra of the Schiff base (HL1)

1HNMR spectrum of the Schiff base

The Schiff base spectrum shows five signals (fig.3) at 11.09, 10.72, 6.88-8.90, 3.321 and 2.499 ppm, downfield of TMS, assignable to the protons of OH (phenyl ring), NH, phenyl ring, DMSO and, CH3, respectively [9-11].

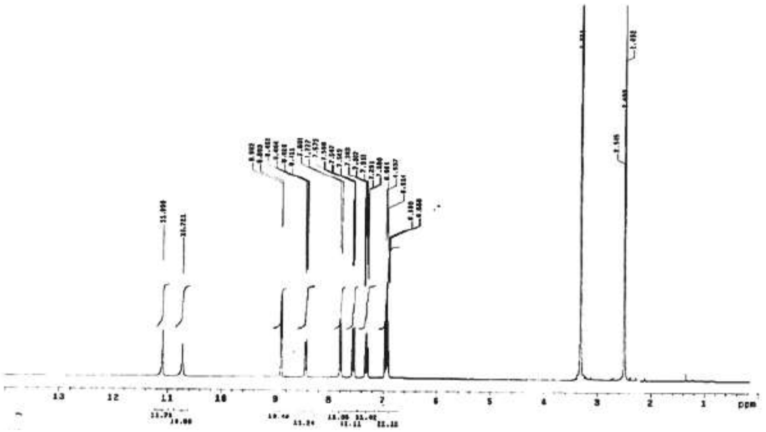


Fig. 3: ^{1}H NMR spectrum of the Schiff base (HL1)

Infrared spectra

The infrared spectra of the Schiff base and mixed ligand chelates (Figs. 4-9) showed bands due to υ(OH), υ(NH) and υ(C=N) respectively, which were absorbed at 3393 cm-1, 3297 cm-1 and 1608 cm-1 [12, 13]. In the IR spectra of mixed ligand complexes: a band at 1608 cm-1 due to υ(C=N) stretching in the spectrum of the Schiff base is shifted to a higher range (1612-1620 cm-1) upon complexation with metal ions via the imine (C=N) nitrogen to the metal center [13-16]. The Schiff base reveals a band at 3393 cm-1 attributed to υ(OH) stretching from the phenolic group which disappears in the spectra complexes indicating the deprotonation of the Schiff base upon chelation and the bands that were offered in the range of 3300-3404 cm-1 corresponded to water molecules [17,18]. Also, new bands in the 437-455 cm-1 region are considered to be due to metal-nitrogen υ(M-N) vibrations, while those accruing around 456-507cm-1 are due to metal-oxygen (M-O) vibrations [19]. The infrared spectrum of Fe(III) mixed ligand chelate shows a band at 1422cm-1 which does not appear in the free Schiff base. This is attributed to the appearance of a nitrate ion group that bonded to the Fe (III) ion through the nitrogen atom [20].

Table 2: Infrared spectral data(cm^{-1}) and electronic spectral transitions of the Schiff base and mixed ligand chelates

Ligand/mixed ligand chelates	IR spectra (cm^{-1})					UV-Vis (cm^{-1})
	$_{\nu}$OH	$_{\nu}$NH	$_{\nu}$C=N	$_{\nu}$M-O	$_{\nu}$M-N	
C14H12N4O5 (HL1)	3393	3297	1608	---	---	π→π*, n→π*
$[Cr(L1)(L2)_2]$	3303	3249	1616	456	437	^{4}A2g(F)→ ^{4}T2g(F) ^{4}A2g (F)→ 4T1g(F)
$[Mn(L1)(L2)(H_2O)_2]$	3404	3300	1612	501	455	6A1g→4T1g(P), CT
$[Fe(L1)(L2)(ONO_2)(H_2O)]$	3300	3100	1615	507	453	$^{3}A_{2g}(F)\rightarrow{}^{3}T_{1g}(F)$,

						${}^{3}A_{2g}(F)\rightarrow{}^{3}T_{1g}(P)$, CT
$[Co(L1)(L2)(H_2O)_2]$	3300	3193	1628	501	453	${}^{4}A_{2g}(F)\rightarrow{}^{4}T_{1g}(F)$, ${}^{4}A_{2}g(F)\rightarrow{}^{4}T_{1}g(P)$
$[Ni(L1)(L2)(H_2O)_2]$	3300	3103	1618	501	453	${}^{3}A2g(F) \rightarrow {}^{3}T1g(F)$, ${}^{3}A2g(F) \rightarrow {}^{3}T1g(p)$

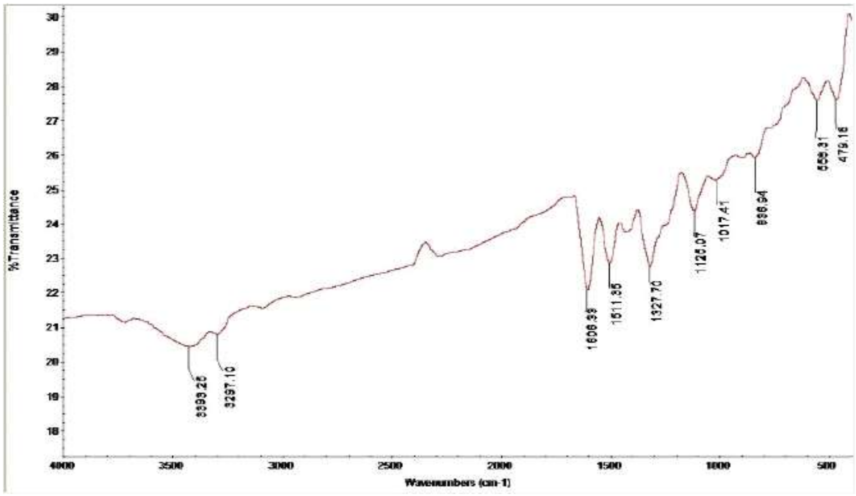


Fig. 4: Infrared spectrum of Schiff base HL1

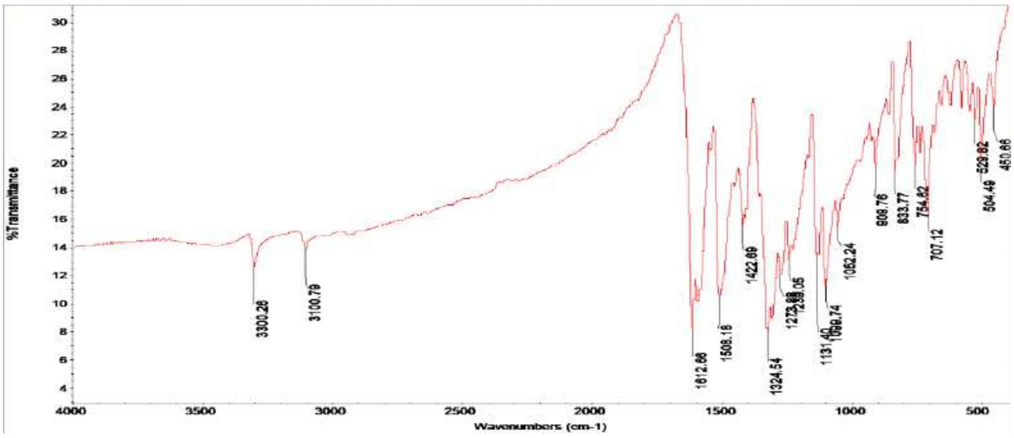


Fig. 5: Infrared spectrum of $[Cr(HL1)(L2)_2]$ mixed ligand chelate

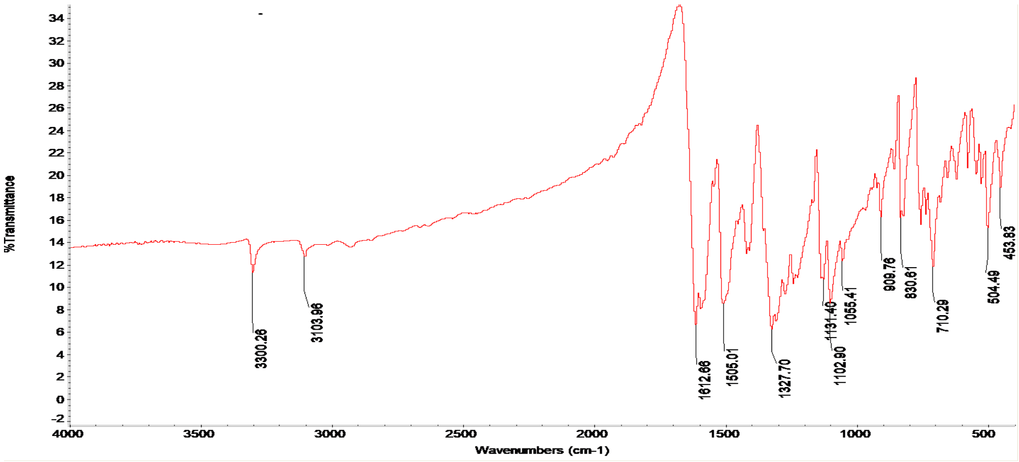


Fig.6: IR spectrum of [Mn(HL1)(L2)(H_2O)$_2$] mixed ligand chelate

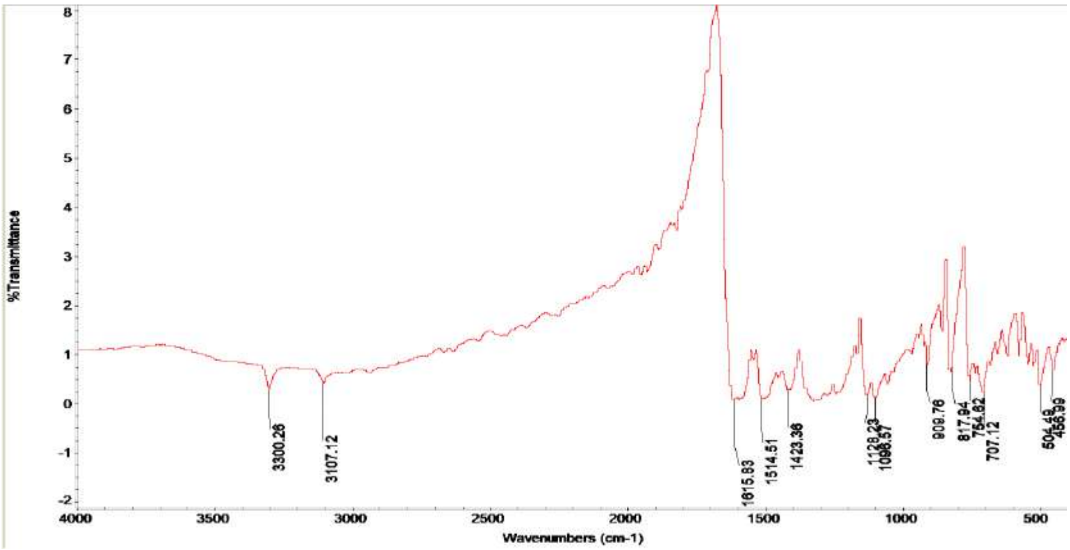


Fig.7: Infrared spectrum of [Fe(HL1)(L2)(ONO_2)(H_2O)]mixed ligand chelate

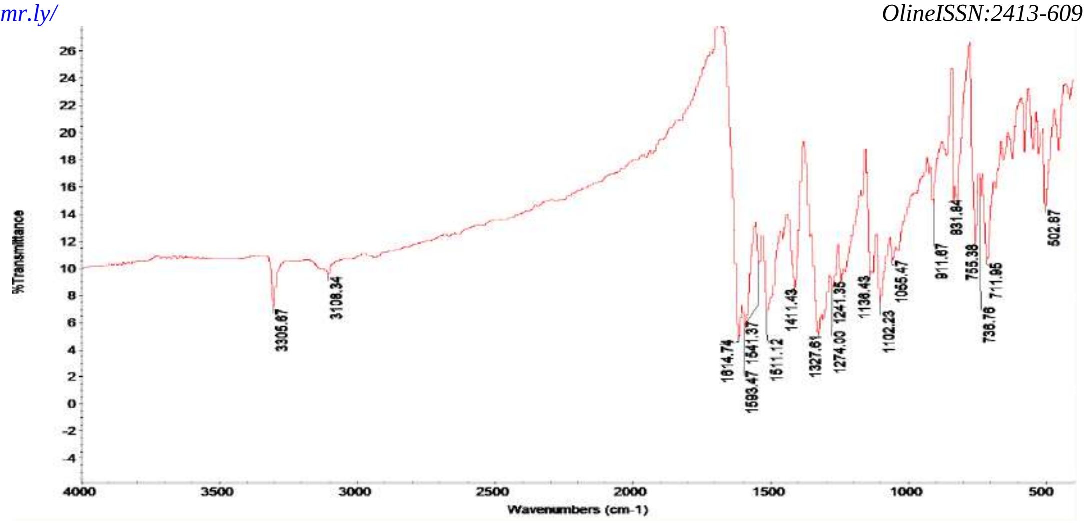


Fig. 8: IR spectrum of [Co(HL1)(L2)(H_2O)$_2$] mixed ligand chelate

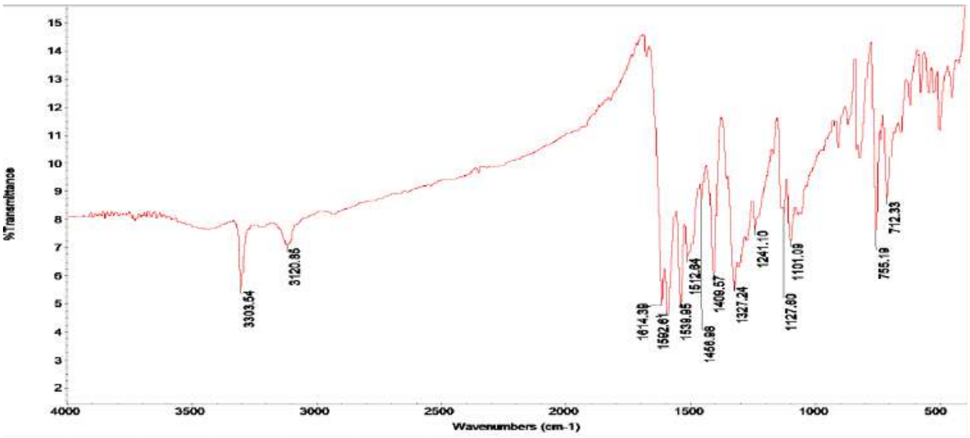


Fig.9: Infrared spectrum of [Ni(HL1)(L2)(H_2O)$_2$] mixed ligand chelate

Electronic spectra and magnetic moments

The electronic spectra of the Schiff base and its mixed ligand chelates are shown in figures [10-15] and their electronic spectral data (cm-1) are listed in table 2. The electronic spectrum of the Schiff base shows bands at 44444 and 28169 cm-1 attributed to π→π* (phenyl ring) and n→π* (C=N) [21, 22]. The magnetic moment value of the Cr (III) chelate (3.78 B. M) and the spectrum of the same chelate which exhibits two bands at 26667 cm-1 and 19802 cm-1 belong to the 4A2g(F)→4T2g(F) and 4A2g(F)→4T1g(F) transitions supporting the presence of an octahedral geometry [23]. Whereas, the spectrum of Mn (II) chelate shows three bands, the first two of them at 18518 and 32258 cm-1 which are due to the 6A1g→4T1g (P) transition and the third one at 35714 cm-1 assigned to the CT transition for an octahedral geometry with a magnetic moment value of 5.65 B.M [24]. The magnetic moment of Fe(III) mixed ligand chelate shows a magnetic moment value of 5.15 B.M confirming the existence of five odd electrons (d5) and the electronic absorption spectrum of this chelate exhibiting three bands at 33333, 28571 and 22471 cm-1 due to the 3A2g(F)→3T2g(F), 3A2g(F)→3T1g(F) and 3A2g(F)→3T1g(P) transitions which suggest an octahedral geometry [25, 26]. The electronic absorption spectrum of Co (II) chelate (3.87B.M) shows two bands at 19230 and 24390 cm-1 due to 4A2g(F)→4T1g(F) and 4A2g(F)→4T1g(P) transitions which confirm an octahedral geometry [26]. While two absorption bands were observed for Ni(II) chelate (2.81B.M) at 20408 and 26667 cm-1 corresponding to 3A2g(F)→3T1g(F) and 3A2g(F)→3T1g(p) transitions, respectively [28].

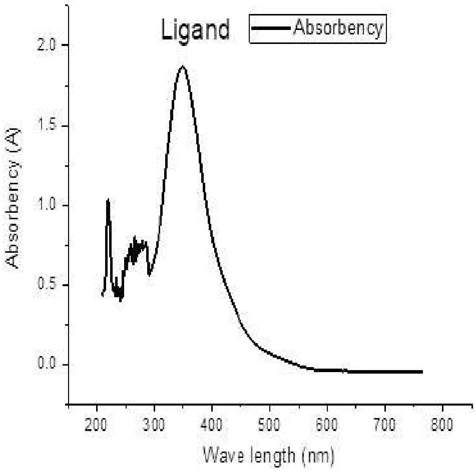


Fig. (10): Electronic spectrum of Schiff base (HL1)

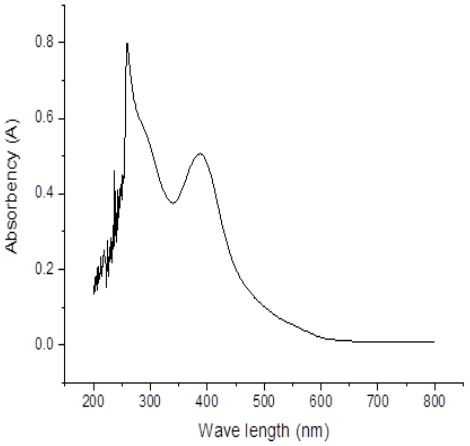


Fig.11: Electronic spectrum of [Cr (L1)(L2)2] mixed ligand chelate

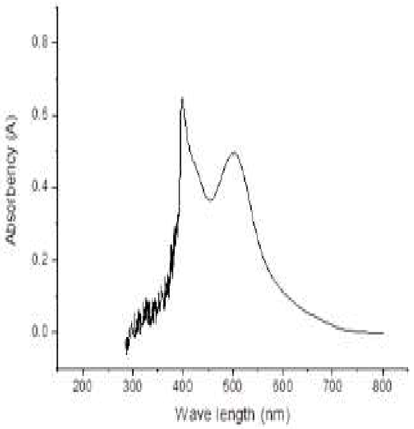


Fig. 12: Electronic spectrum of [Mn(L1)(L2)(H2O)2] mixed ligand chelate

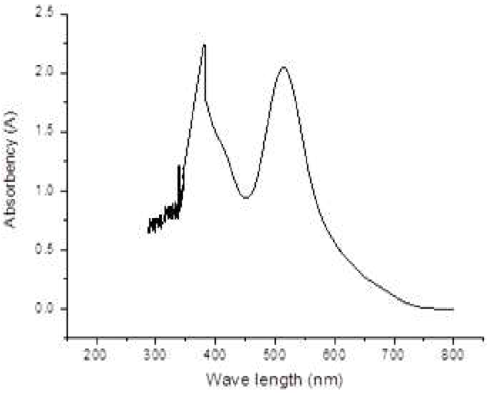


Fig. 13: Electronic spectrum of [Fe(L1)(L2)(ONO2)(H2O)] mixed ligand chelate

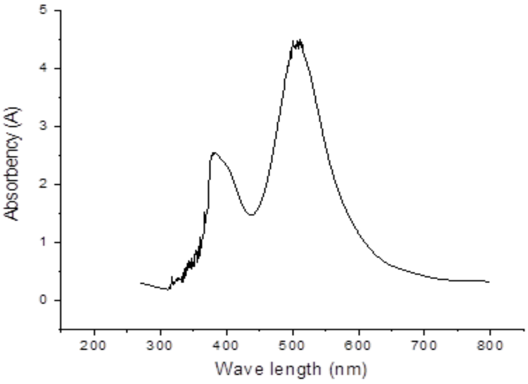


Fig. 14: Electronic spectrum of [Co(L1)(L2)(H2O)2] mixed ligand chelate

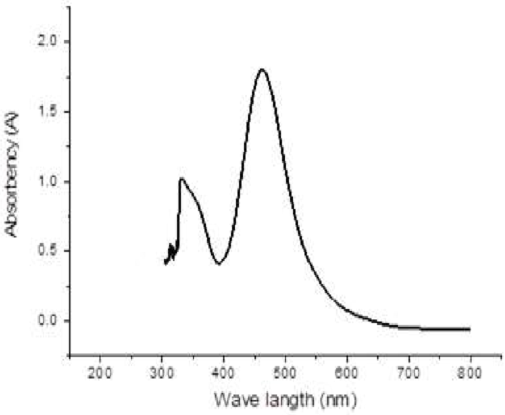


Fig. 15: Electronic spectrum of [Ni(L1)(L2)(H2O)2] mixed ligand chelate

Thermogravimetric analysis

The thermogravimetric analysis (weight % against temperature) opens an acknowledgment of the existence of water molecules as hydrated or coordinated in the complex in addition to the decomposition of the used ligand and the formation of the metal oxide at the last step [29,30]. The thermogram curve of the MnL1L2 mixed ligand complex (Table 3, fig.16) shows three steps: The first step represents the removal of two coordinated water molecules at a temperature range of 22.86-194.460C. The second and third steps belong to the decomposition of the Schiff base and 2-aminobenzoic acid ligands at a temperature of the range 194.46-407.460C [31,32]. Whereas at a temperature of 530.30-8000C, a residue of MnO appeared at 13.620% by weight. The Ni(II) mixed ligand complex curve(Fig.17) shows a weight % of 6.16(Table 3) which is attributed to the removal of two water molecules at a temperature of 179.19-322.030C, The weights% of 24.713 and 54.854 are assigned to the decomposition of the Schiff base in two steps at a temperature range of 322.03-550.240C. Meanwhile, the weight % of 13.822 corresponds to the formation of metal oxide (NiO) at a temperature over 550.240C[33,34].

Table 3: Thermogravimetric analysis of some mixed ligand complexes

Compound	Decomposition Steps	Temperature range (°C)	Remove species	Wt. loss	
				% (Cal.)	% (Found)
[Mn(L1)(L3)(H_2O)$_2$]	1st	108.94-250.22	$(H_2O)_2$	6.640	6.629
	2nd	250.22-427.15	$C_7H_6NO_2$	25.100	25.099
	3rd	427.15-530.40	$C_{14}H_{11}N_4O_4$	55.180	55.205
	Residue	>530.40	MnO	13.080	13.067
[Ni(L1)(L3)(H_2O)$_2$]	1st	179.19-322.03	$(H_2O)_2$	6.592	6.612
	2nd	322.03-431.73	$C_7H_6NO_2$	24.926	24.713
	3rd	431.73-550.24	$C_{14}H_{12}N_4O_4$	54.799	54.854
	Residue	550.24-800.24	NiO	13.676	13.822

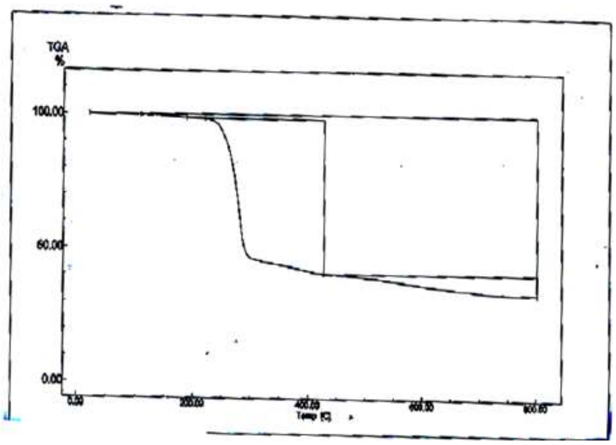


Fig. 16: TG of MnL1L2 mixed ligand chelate

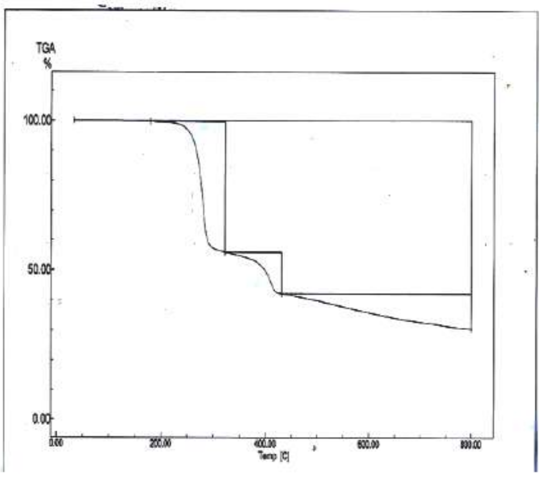


Fig.17: TG of NiL1L2 mixed ligand chelates

Antibacterial activity

The antibacterial efficacy of various mixed ligand complexes was assessed through their inhibition zones against *Staphylococcus aureus* and *Escherichia coli*. The results, measured in millimeters and expressed as the mean ± standard deviation (SD) $[Cr(L1)(L2)_2]$ Complex exhibited the highest antibacterial activity among the tested compounds. Against *Staphylococcus aureus*, it showed an inhibition zone of 15±0.8 mm, and against *Escherichia coli*, the inhibition zone was 13±0.7 mm.$[Mn(L1)(L2)(H_2O)_2]$ and $[Ni(L1)(L2)(H_2O)_2]$ Complexes Both of these complexes demonstrated no antibacterial activity against either of the tested bacteria. The inhibition zones for both *Staphylococcus aureus* and *Escherichia coli* were recorded as 0.0 mm, indicating a lack of efficacy $Fe(L1)(L3)(NO_3)(H_2O)]$ Complex showed moderate antibacterial activity. Against *Staphylococcus aureus*, it had an inhibition zone of 8±0.6 mm, while against *Escherichia coli*, the zone was slightly smaller, measuring 7±0.5 mm $[Co(L1)(L2)(H_2O)_2]$ Complex Similar to the $[Fe(L1)(L3)(NO_3)(H_2O)]$ complex, this compound displayed moderate antibacterial properties. It inhibited *Staphylococcus aureus* with an inhibition zone of 8±0.6 mm and showed relatively higher activity against *Escherichia coli* with an inhibition zone of 11±0.5 mm. The antibacterial results (mm) are presented in Table 4.

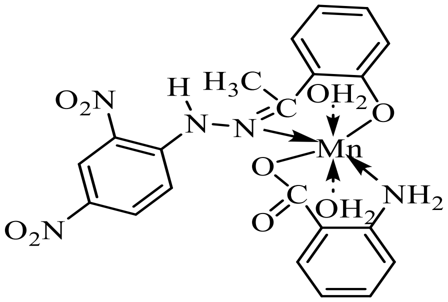
Table 4: Antibacterial activity results (mm) of the mixed ligand chelates

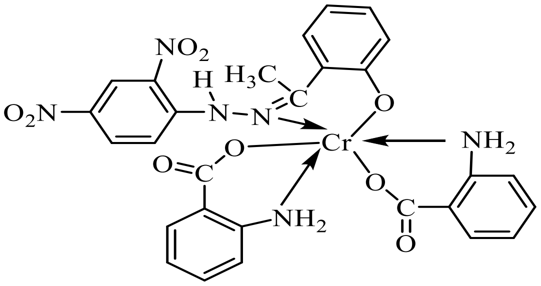
Mixed Ligand complexes	**Bacteria/ inhibition zone (mm / Mean ± SD)**	
	Staph. Aures	*E. coli*
$[Cr(L1)(L2\ _2]$	15±0.8	13±0.7
$[Mn(L1)(L2)(H_2O)_2]$	0.0	0.0
$[Fe(L1)(L3)\ (NO_3)\ (H_2O)]$	8±0.6	7±0.5
$[Co(L1)(L2)(H_2O)_2]$	8±0.6	11±0.5
$[Ni(L1)(L2)(H_2O)_2]$	0.0	0.0

L_1 = Schiff base L_2 = 2-aminophenol L_3 = 2-aminobenzoic acid

Conclusion

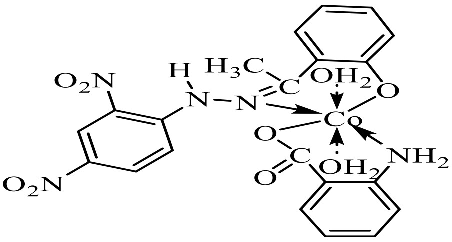
Based on the results obtained from the analytical and spectroscopic tools, an octahedral structure was suggested for all the chelates as shown below:

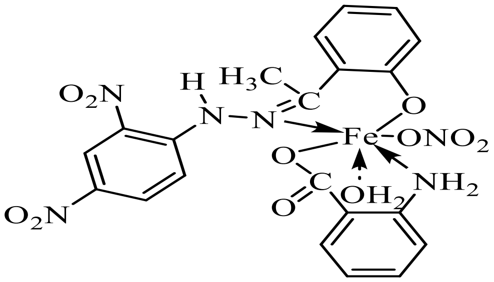




Structure of $[Cr(L1)(L2)_2]$ mixed ligand chelate

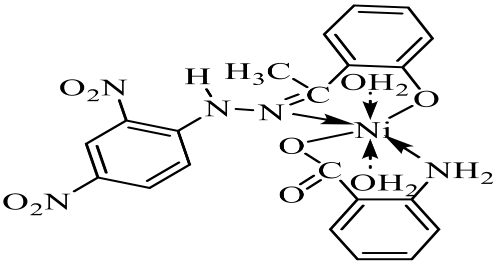
Structure of $[Mn(L1)(L2)(H_2O)_2]$ mixed ligand chelate





Structure of $[Fe(L1)(L2)(ONO_2)(H_2O)]$ mixed ligand chelate

Structure of $[Co(L1)(L2)(H_2O)_2]$ mixed ligand chelate



Structure of $[Ni(L1(L2)(H_2O)_2]$ mixed ligand chelate

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