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Original article

Dual Ligand Chelates: Preparation and Spectroscopic Characterization

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Abstract
Here, we synthesized three new blended ligand complexes of chromium (III), iron (III), and lanthanum (III) ions with a Schiff base made from the condensation of [o-aminophenol and 2-hydroxyacetophenone in the presence of concentrated sulphoric acid (HL1)] as a primary ligand and o-nitroaniline (L2) as a secondary. The Schiff base and its dual ligand chelate were characterized using several spectroscopic studies, IR, ^{1}HNMR, electronic and mass spectra, in addition to elemental analyses, molar conductivity measurements, and magnetic moments. The spectroscopic and analytical outcomes confirmed the formation of the chelates in a 1:1:1(L1: M: L2) ratio. Similarly, an octahedral structure became counseled for all chelates.
Keywords: Schiff Base, O-aminophenol, 2-hydroxyacetophenone, O-nitroaniline, Dual Ligand Chelates.

Introduction

Schiff bases are biologically important compounds involved in diverse tactics fashioned after intramolecular dehydration and have the fashionable formulation R1R2C=N – R3, wherein R3 is an aryl or alkyl group [1]. Schiff bases of aliphatic aldehydes are incredibly volatile and with no trouble polymerizable, even as the ones of fragrant aldehydes having an effective conjugation, and are extra stable. Ternary complexes, typically referred to as dual ligand complexes with exclusive ligands coordinated to the identical crucial metallic ion, were widely used in analytical chemistry [2-4]. Recent studies have proven that the ternary complexes with biologically effective ligands facilitate utilization in various enzymatic reactions and numerous organic processes. Schiff base, a commonly used natural ligand, has performed an essential role in the increase of coordination chemistry because of its ease in synthesis, chelating behavior, balance in numerous oxidative and reductive situations, and structural similarities with herbal biological substances [5,6]. Alassbaly et al., Prepared five mixed ligand chelates of Cr (III), Co (II), Ni (II), Zn (II) and Cu (II) ions with a Schiff base of 4-dimethylaminobenzaldehyde with 2-aminophenol "as main ligand", L-Histidine as co-ligand by physiochemical techniques for prepared mixed ligand chelates supported that an octahedral geometry for all complexes [7-10].
Dual ligand chelates prepared from di-and trivalent metal ions [Cr, Co, Ni, and Zn ions], and Schiff base (HL1) resulted from the condensation of 4-dimethylaminobenzaldehyde with 2-aminophenol as primary ligand, whereas anthranilic acid (L2) represented the secondary ligand. The prepared Schiff base and its dual ligand chelates have been characterized by using several analytical and spectroscopic tools. The chelates showed antimicrobial activity on different species of pathogenic bacteria.
The following study concerned the preparation and characterization of a Schiff base as a primary ligand and o-nitroaniline as a secondary ligand to fit the geometrical structures of trivalent metal ions (Cr, Fe, La) dual ligand chelates.

Material and Methods

All chemical compounds used in this research were of pure grade (BDH or Aldrich). Include: o-aminophenol, 2-hydroxyacetophenone, Conc.H_2SO_4, DMF, o-nitroaniline, DMSO, NaOH, methanol, ethanol, $FeCl_3.6H_2O$, $CrCl_3.6H_2O$, La $(NO_3)_3.6H_2O$, and distilled water. The CHN analyses for the synthesized compounds had been made on a 2400-CHN elemental analyzer. The molar conductivity was determined in DMF solvent on the CMD-650 virtual conductivity meter, Benghazi University. The infrared spectra have been obtained on an IFS-25 DPUS/I spectrometer. The ^{1}HNMR spectra had been recorded on a Varian Gemini 2 hundred–200 MHz spectrometer using TMS as an internal standard in d6-DMSO. The electronic and mass spectra have been recorded on a Perkin-Elmer Lambda-365 spectrophotometer and a Shimadzu QP-2010 Plus spectrometer, respectively.

Synthesis of Schiff Base (HL1)

A warm methanol solution of o-aminophenol (1.09 g, 0.01mol) and 2-hydroxyacetophenone (1.39g, 0.01 mol) was jumbled together in the presence of concentrated sulphoric acid and refluxed for three hours on a hot plate with a magnetic stirrer. The ensuing combination was gathered by using filtration and dried in air. The obtained product was recrystallized with absolute methanol to obtain yellow crystals [11]. The chemical structure of the synthesized Schiff base under research is proven in (Scheme 1).

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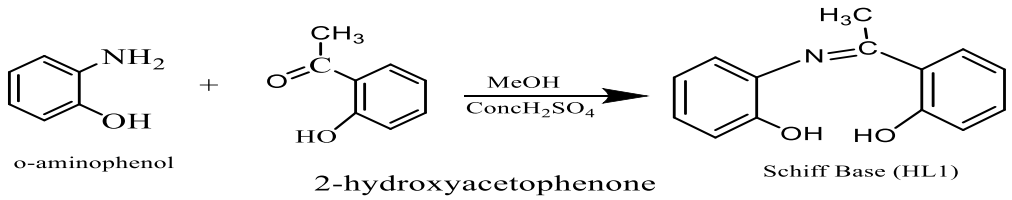
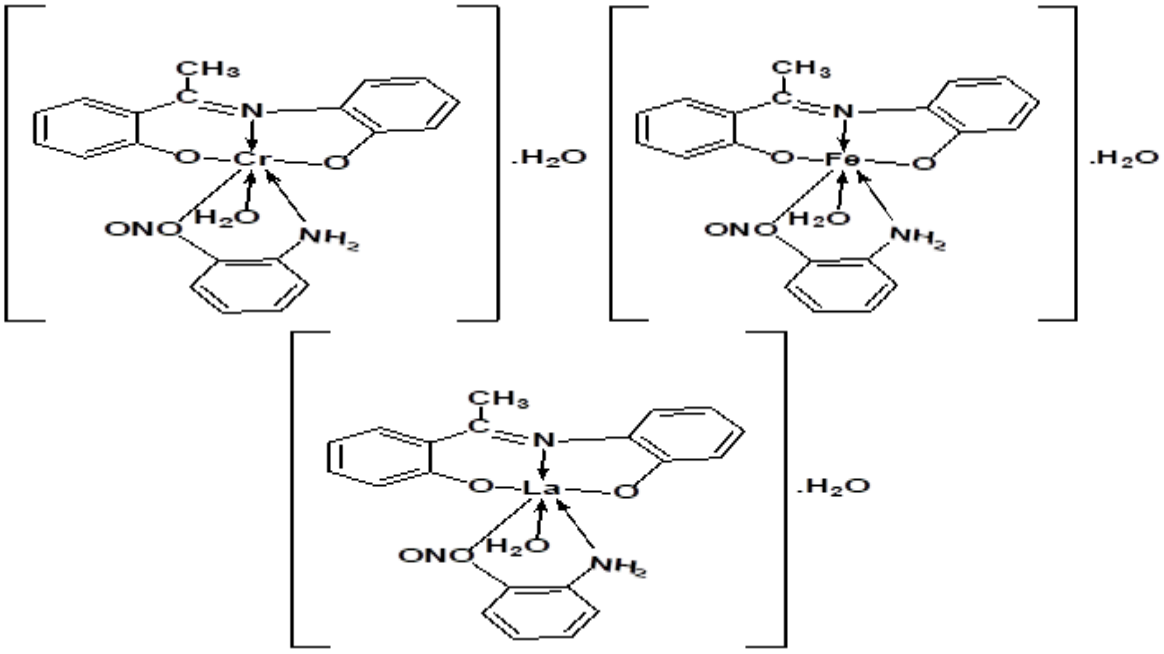


Scheme 1. Synthesis of the Schiff base (HL1).

Synthesis of the dual ligand chelates

The dual ligand complexes of these ligands were prepared by way of refluxing a methanolic solution of Schiff base (2.28 g, 0.01 mole) and the same amounts of the metallic salts [$FeCl_3.6H_2O$; 2.70g, $La(NO_3)_3.6H_2O$; 4.3g, and $CrCl_3.6H_2O$; 2.67g] for 2 hours. Few drops of 10% sodium hydroxide answer were delivered slowly to adjust the pH fee at 8 until the chelates separated, and then the secondary ligand, o-nitroaniline (L2) (0.01 mole, 1.38g) in 25cm^3 of absolute methanol turned into added dropwise. The merchandise had been refluxed again for an additional 3 hours. The obtained products were filtered and washed several times with warm ethanol until the filtrates became clear. The resulting chelates have been dried in a desiccator below calcium chloride [12].



Scheme 2. The suggested structures of dual ligand complexes.

Results and Discussion

Elemental analysis and molar conductance measurements

The CHN elemental analysis statistics of the Schiff base and its dual ligand chelates are given in (Table 1). These analyses are partially useful in determining the empirical system of the compounds. The experimental outcomes are in great agreement with the calculated ones and monitor that the chelates are fashioned in 1:1:1 [M: L1: L2] ratios. The molar conductance values of the dual ligand chelates (zero values) monitor the lifestyles of non-electrolytic nature for all of the chelates, indicating that they are pure [13].

Table 1. Elemental analysis data and some physical properties of Schiff base and dual ligand complexes are cited.

Ligand-complexes	M.Wt	Color	C% (Calc.) Found	H% (Calc.) Found	N% (Calc.) Found	(BM)μ
Schiff base (HL1)	228	yellow	(73.68) 73.20	(5.70) 5.94	(6.14) 6.54	--------
[Cr (L1) (L2)(H_2O)] H_2O	451	Black	(44.44) 44.70	(3.70) 3.48	(7.77) 7.65	4.49
[Fe (L1) (L2) (H_2O)] H_2O	543.85	Carob	(44.12) 43.90	(3.67) 3.44	(7.72) 7.44	5.74
[La (L1) (L2) (H_2O)] H_2O	626.91	Hickory	(38.28) 38.40	(3.19) 2.95	(6.69) 6.45	0.00

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Infrared spectra

The infrared spectral facts of the Schiff base and its dual ligand chelate have been listed in (Table 2) and their spectra have been given in (Figures 1-5). The infrared spectra of the dual ligand chelates show off bands inside the range of 3450-3480 $cm^{-1,}$ analogous to the presence of water molecules [14]. Meanwhile, the identical spectra show bands within the range of 1520-1570 cm^{-1} assigned to v(C=N) vibration, the changing of these bands in relation to the Schiff base (HL1) confirmed the participation of this institution in chelation via nitrogen atom [15]. The other coordination websites that can take part in coordination are NH_2 (3300 cm^{-1}) and NO_2 (1550 cm^{-1}) agencies of the ligand (L2). The $–NH_2$ organization band in the spectra of Fe (III) and La (III) dual ligand chelates overlaps with the bands of water molecules [16]. But inside the Cr (III) L1L2 dual ligand chelates, this institution is changed, confirming the participation of amine institution in complexation with metallic ions [17]. In addition, the change of the NO_2 institution band of o-nitroaniline within the spectra of the chelates suggests the involvement of this organization in bonding with the metal ions via the oxygen atom [18]. New bands inside the variety of 610-640 and 450-475 $cm^{-1,}$ which aren't gift in the loose ligands, are due to v(M-O) and v(M-N) vibrations, and their appearance helps the entering of oxygen and nitrogen atoms of the C=N, $-NO_2$ and –OH organizations of the unfastened ligands in complexation technique [19].

Table 2. Infrared frequencies (cm^{-1}) of the Schiff base and its dual ligand chelates.

Ligand-complexes	ᵛOH	ᵛC=N	ᵛM-O	ᵛM-N
Schiff base (HL1)	3875	1687	-	-
[Cr (L1) (L2) (H_2O)] H_2O	3450	1540	610	450
[Fe(L1) (L2) (H_2O)] H_2O	3455	1570	640	475
[La(L1) (L2) (H_2O)] H_2O	3480	1520	620	460

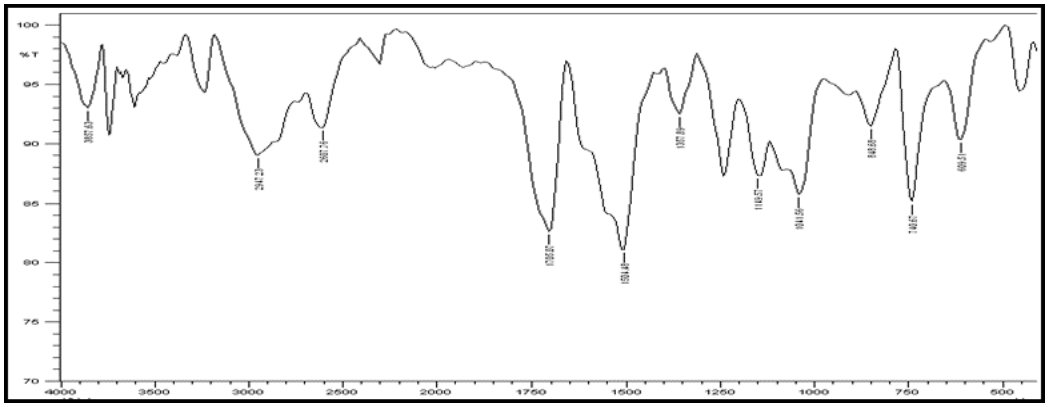

Figure 1. Infrared spectrum of Schiff base (HL1).

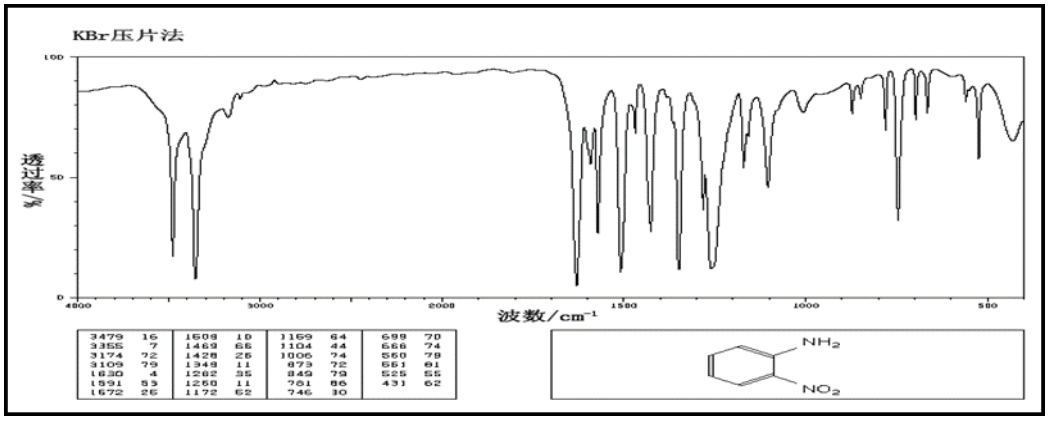

Figure 2. Infrared spectrum of o-nitroaniline.

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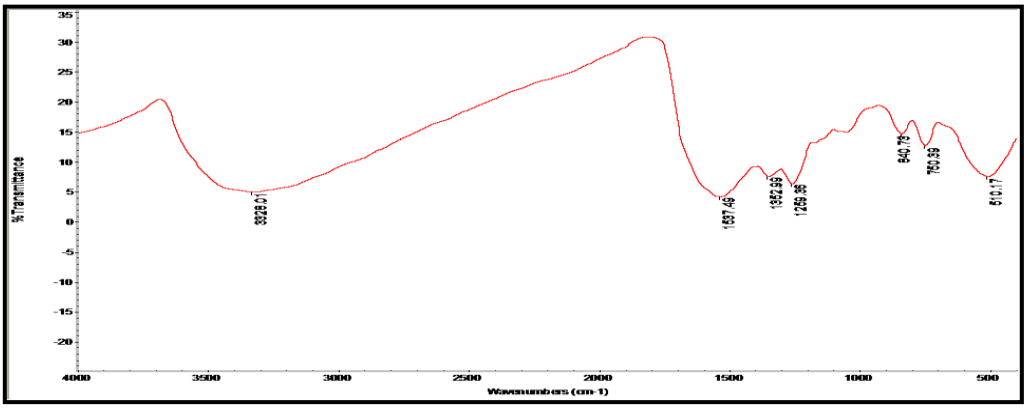

Figure 3. Infrared spectrum of Cr (III) dual ligand complex.

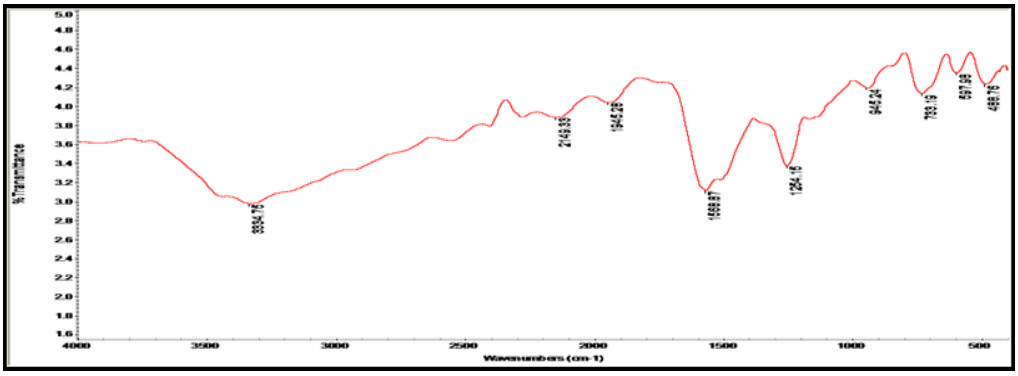

Figure 4. Infrared spectrum of Fe (III) dual ligand chelate.

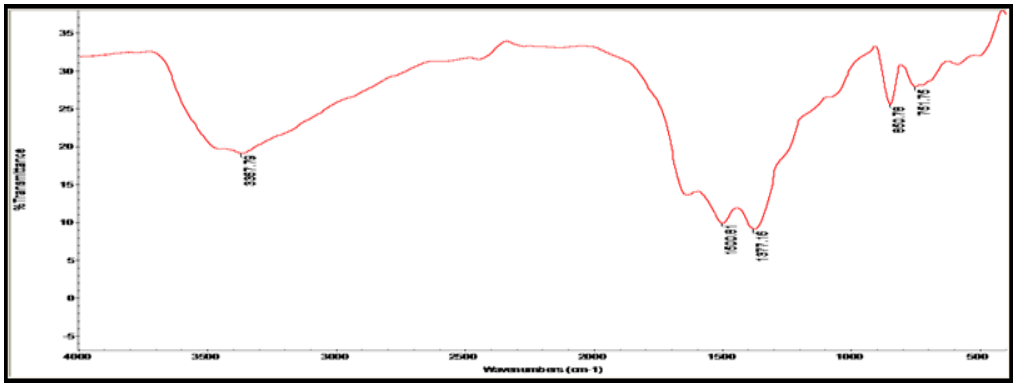

Figure 5. Infrared spectrum of La (III) mixed ligand complex.

[1]HNMR Spectra of Schiff base (HL1)

The [1]HNMR spectrum of the Schiff base (HL1) shows alerts within the range 6.50-6.77 ppm are assigned to the presence of protons of the phenyl ring moiety. The signals at 7.80-7.90 ppm, which are attributed to -OH agencies of the Schiff base (HL1) (Figure 6). The methyl institution and DMSO solvent are seen at 2.35 and 2.75ppm, respectively.

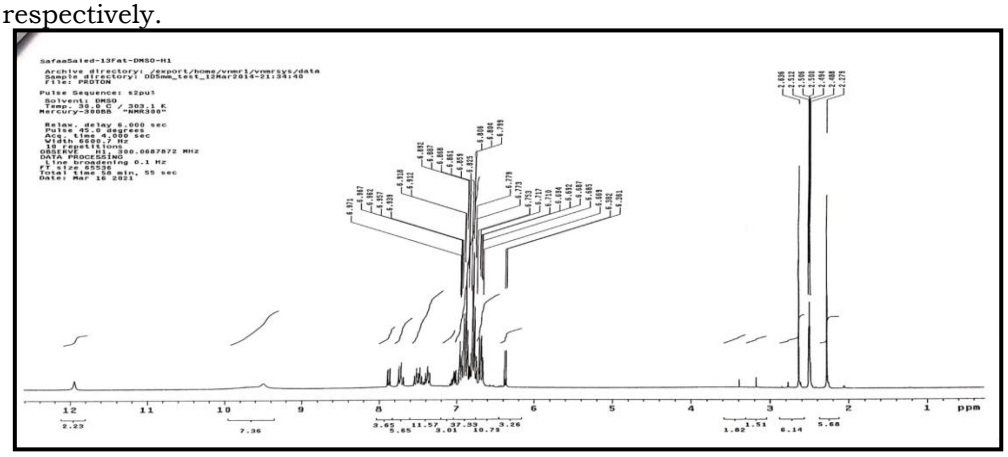

Figure 6. [1]HNMR spectrum of Schiff base (HL1).

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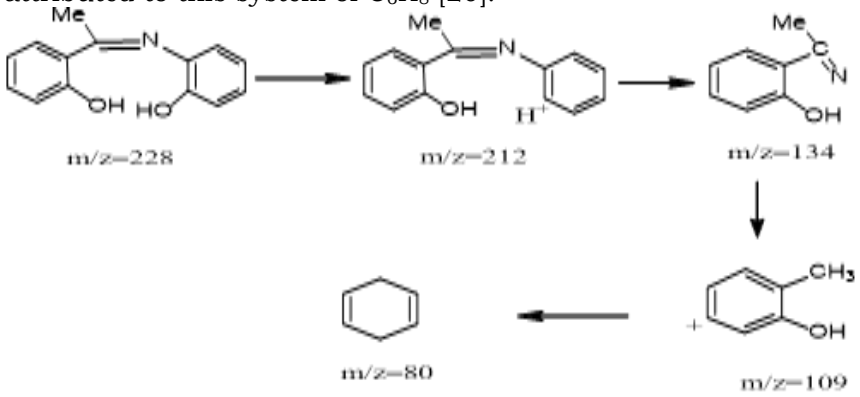
Mass spectral fragmentation of Schiff base (HL1)

The mass spectral fragmentation of the Schiff base (HL1) is proven in (Scheme 2), (Table 3), and (Figure 7). The base height at m/z⁺ = 228 due to the unique method of some other height at m/z+=134 is equal to the formula of $C_8H_8NO^+$. The structure $C_7H_7O^+$ has similarities to the peak at m/z+=109. Meanwhile, the final peak at m/z+=80 is attributed to this system of C_6H_8 [20].



Scheme 3. Mass spectral fragmentation of Schiff base.

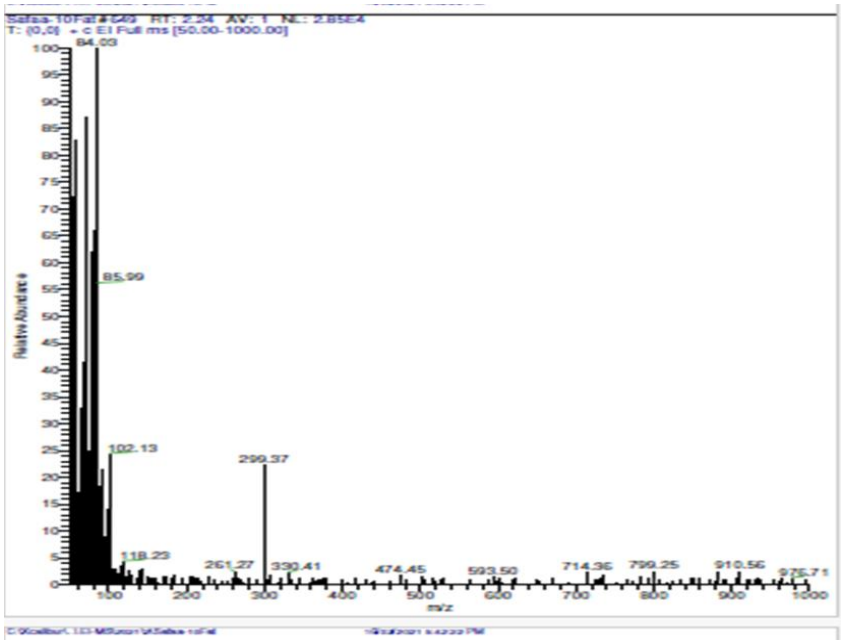


Figure 7. Mass spectrum of Schiff base ligand (HL1)

Table 3. Mass spectral fragmentation of the Schiff base and mixed ligand chelate.

Compound	Fragmented ions	m/z⁺ values
$C_{14}H_{13}NO_2$(HL1)	$C_{14}H_{13}NO_2$	228
	$C_{14}H_{13}NO^+$	212
	$C_8H_8NO^+$	134
	$C_7H_7O^+$	109
	C_6H_8	80

Electronic spectra and magnetic moments

The ultraviolet spectra of Schiff base and mixed ligand chelates are listed in (Table 4) and their spectra are shown in (Figures 8-11). (Figure 8) became recorded at ambient temperature in 10^{-3} M DMF solvent and shows bands at 289nm (34602cm^{-1}) and 310 nm (32258 cm^{-1}), attributing to π-π (phenyl ring) and n-π transitions [21,22]. The digital spectrum of Cr (III) chelate (Figure 9) displays bands at 275nm (36363cm^{-1}) and 380nm (26315cm^{-1}) assigned to 4A2g→4T2g, 4A2g→4T1g(F) transitions [23,24]. The magnetic moment of Cr (III) chelate is 4.49 B.M., which is in accordance with the observed spin -best fit, suggesting an octahedral environment around the chromium ion [25]. The electronic spectrum of Fe (III) chelate (Figure 10) presents two bands at 278nm (35971cm^{-1}) and 387nm (25839cm^{-1}) assigned to $^6A_1g\rightarrow{}^4T_1g(4D)$, 6A1g→4T1g(4A2) transitions [26]. The magnetic moment of Fe (III) is 5.74 B.M., confirming the lifestyles of excessive spin Fe (III) complex [27]. The digital spectrum of La (III) chelate (Figure 11) indicates two bands at 378nm (26455cm-1) and 396nm (25252cm^{-1}) attributed to π-π*, n-π*, and LMCT transitions [28,29].

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Table 4. Electronic spectral data (nm, cm^{-1}) of ligands and their dual ligand chelates.

Ligand /Chelate	nm(cm^{-1})
Schiff base (HL1)	289(34602),310(32258)
o-nitroaniline	310(32258),415(24096)
[Cr(L1)(L2)(H_2O)] H_2O	275(36363),380(26315)
[Fe(L1)(L2)(H_2O)] H_2O	278(35971),387(25839)
[La(L1)(L2)(H_2O)] H_2O	378(26455),396(25252)

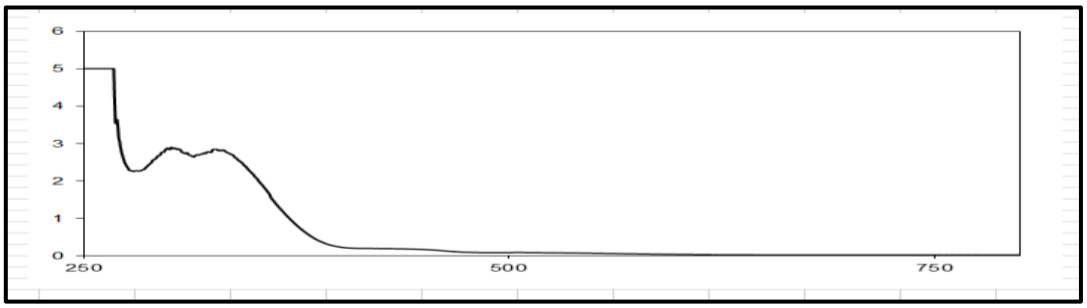

Figure 8. Electronic spectrum of Schiff base (HL1).

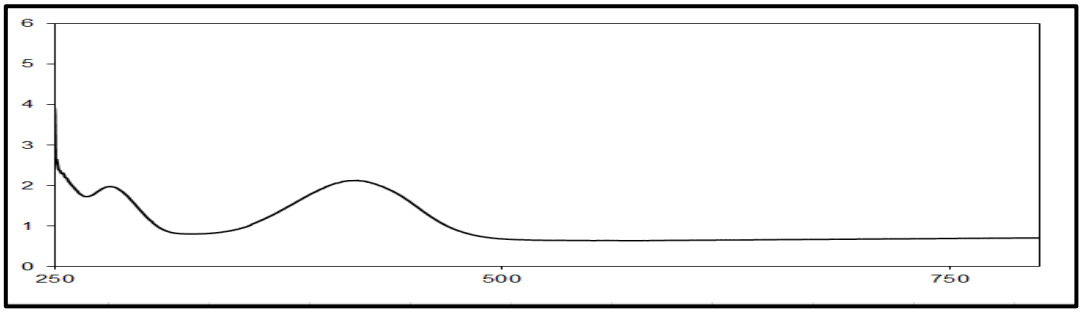

Figure 9. Electronic spectrum of Cr (III) dual ligand chelate.

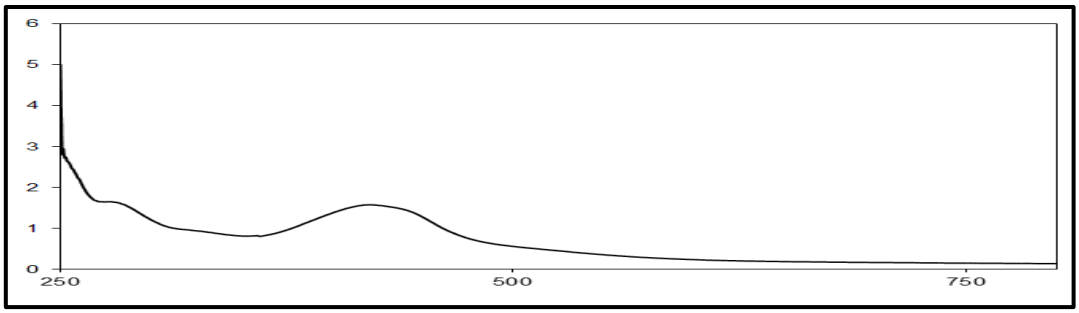

Figure 10. Electronic spectrum of Cr (III) dual ligand chelate.

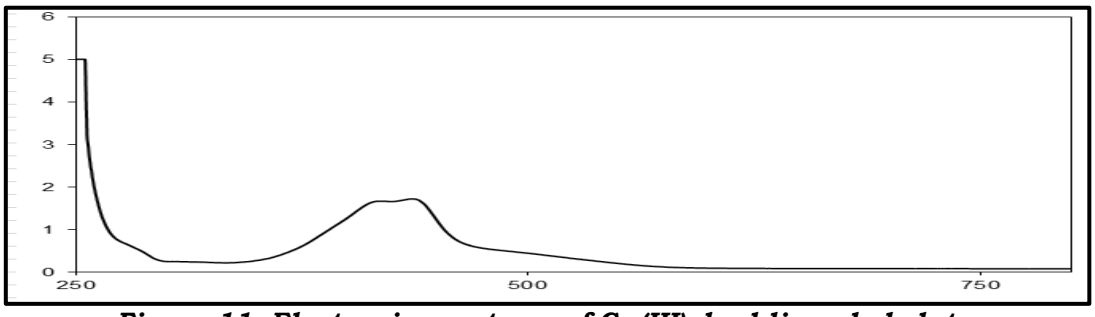

Figure 11. Electronic spectrum of Cr (III) dual ligand chelate.

Conclusion

The analytical and spectral investigations confirm the successful formation of the Schiff base ligand and its corresponding mixed-ligand metal chelates. The results indicate that the metal ion coordinates with both ligands to form stable chelates. All synthesized complexes display an octahedral geometry, as supported by the observed spectral features and coordination behavior. These findings collectively verify the formation of the intended structures and highlight the consistency between analytical data and the proposed geometries.

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Conflict of interest. Nil

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