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Article

Synthesis, characterization and thermal study of the new Schiff base ligand derived from sulfonic acid with Cobalt, Nickel, Zinc and cadmium complexes

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Abstract

We study in the presented work Schiff base formation from reaction of 4-amino-3-naphthol-1-sulfonic acid with 3-chioro-borobenzaldehyde. Common organic compounds of major relevance in coordination chemistry are Schiff bases since they can combine with metal ions for the purpose of generating metal complexes. The Schiff base has been formed then reacted with certain transition metal chlorides including nickel, cobalt, cadmium, and zinc. This reaction produced unique metal complexes with particular qualities. The characteristics and structure of such complexes were ascertained by means of numerous advanced analytical approaches. Among such methods were infrared (IR) spectroscopy, which enabled the ligand-metallic element chemical bond identification. Additionally utilized to investigate light absorption and extract information on the electronic interactions in the complexes is ultraviolet (UV) spectroscopy. Furthermore examined internal structure regarding the complexes was nuclear magnetic resonance (NMR) spectroscopy. At last, the stability of complexes under various temperatures has been investigated with the use of the thermogravimetric (TGA) analyses. The work intends to generate complexes with physical and chemical characteristics suitable for use in materials science, analytical chemistry, and environmental chemistry as well as to deepen knowledge of Schiff base interactions with the metal ions.

Keywords: azomethine, sulfonic, thermogravimetric

Introduction

The azomethine (-CH=N-) functional group develops from reflux reaction between a ketone or aldehyde and a main amine, therefore defining Schiff base composites [1]. Research show the azomethine group functions as an electron donor as well as an acceptor. It serves as a donor through non-bonding electron pair on nitrogen atom and as acceptor through double bond's π orbital [2]. Usually produced from benzaldehyde or its derivatives, Schiff bases are combined with aniline or its derivatives. Those molecules show structural asymmetry since benzene ring linked to atom of nitrogen is orientated outside the plane of the remainder of molecule. Moreover, it was seen that substituents on benzaldehyde moiety affect electronic distribution more than those on the aniline ring [3].

In many disciplines, especially pharmacology, in which they have antibacterial, antifungal, and anticancer effects [4], Schiff bases have become rather important. They also find use in industry, notably as heat initiators for radical polymerization techniques [5].

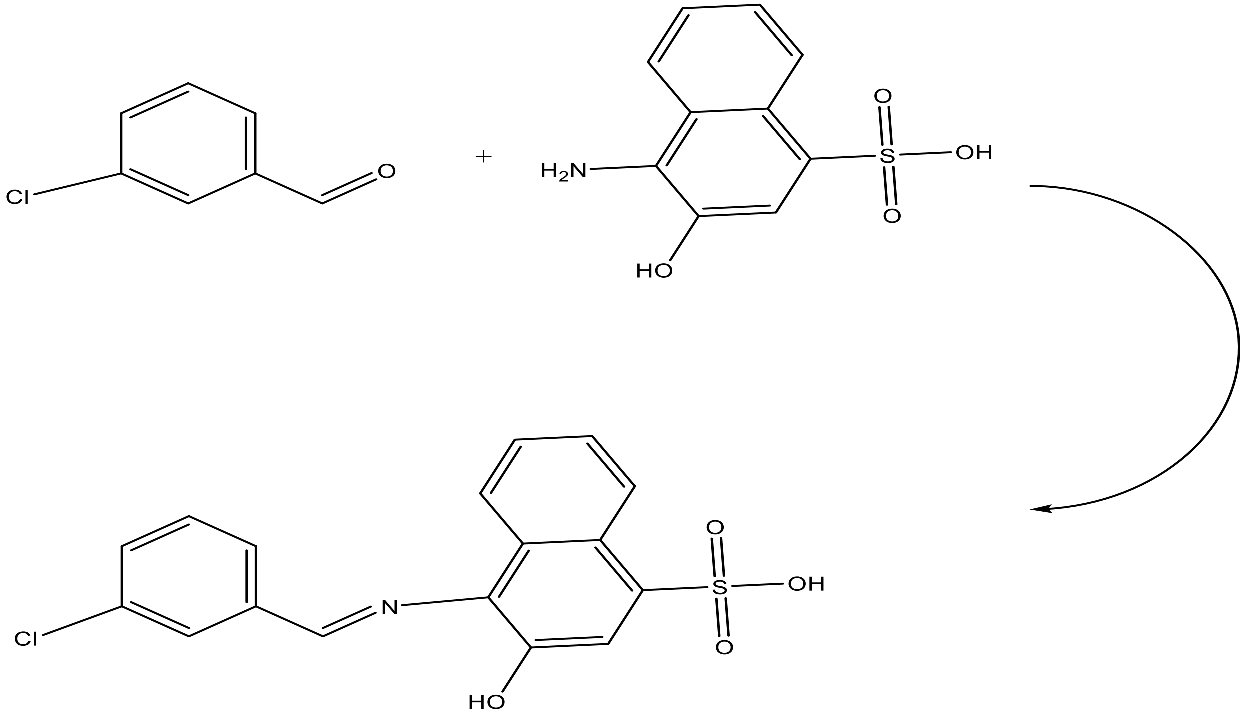
Instrumentation

Samples were characterised by thermogravimetric analysis (TGA). With the use of potassium bromide (KBr), Fourier-transform infrared (FT-IR) spectra have been obtained with Shimadzu 8400S spectrometer between 400 cm^{-1} and 4000 cm^{-1}. Furthermore examined were ^{1}H-NMR spectra, and elemental analysis (CHNS) with a Euro-EA Elemental Analyzer.

Schiff Base Ligand Synthesis

Preparation of (E)-3-Chlorobenzaldehyde-(amino)-naphthalene-1-sulfonic acid

A round-bottom flask was used to dissolve 3-chlorobenzaldehyde (2.06mmol, 0.29g) in 15mL of ethanol, followed by the addition of 1amino-2-naphthol-4-sulfonic acid (2.08mmol, 0.5g) dissolved in 15mL of the ethanol. The reaction mixture has been subjected to reflux for 5 hours [6]. After completion, the flask has been left in an ice bath for a short period, and the resulting precipitate has been filtered then dried after that, yielding a pale pink solid Every composite used was of best quality (BDH, Fluka). The synthesis of the Schiff base is depicted in Scheme1.



Scheme1: preparation of the Schiff base Ligand

Preparing metallic complexes [Co(Ⅱ), Ni(Ⅱ), Zn(Ⅱ), Cd(Ⅱ)] via ligand (L)

0.55mmol (0.2g) of Schiff base was dissolved in 10 mL of solvent(ethanol) and then added to the KOH that had been dissolved in 10mL of absolute solvent, thereby arranging chelate facilities of the ligand and the metal (1:2). Liquefied in absolute ethanol 10mL varying with ligand and refluxed for 3hr on contents cooling, consistent hydrated metal chloride salt of $CoCl_2$. 6 H_2O=0.065g, $NiCl_2.6H_2O$=0.065g, $ZnCl_2$=0.037g and $CdCl_2.H_2O$=0.55g was liquefied. The facilities have been divided out in each one of the events. The produce was filtered, solvent-splashed, after that dehydrated under vacuum.

Results and discussion

Elemental analysis

Aiming at determining the chloride content, a few of physical and analytical properties of ligand and ligand complexes have been investigated.

Table1: Physical properties and analytical data of ligands and their complexes

Comp.	**Chemical Formula**	**M.wt.**	**MP**	**color**	**Analysis. Calc | (Found)**						
					M	**C**	**H**	**N**	**S**	**O**	**Cl**
L	$C_{17}H_{11}NO_4SCl$	361.8	194-196	Pala pink	-	56.38	30.4	3.86	8.84	17.68	9.81
					(-)	(56.35)	(30.02)	(3.84)	(8.41)	(17.69)	(9.80)
1	$[CoC_{34}H_{28}N_2O_{11}S_2Cl_2]$	833.9	292-294	Purple	7.06	48.92	3.35	3.35	7.67	21.10	8.51
					(7.01)	(48.90)	(3.33)	(3.31)	(7.64)	(21.06)	(8.48)
2	$[NiC_{34}H_{28}N_2O_{11}S_2Cl_2]$	833.6	295-297	brown	7.04	48.94	3.35	3.35	7.67	21.11	8.51
					(7.00)	(48.91)	(3.32)	(3.33)	(7.65)	(21.08)	(8.49)
4	$[ZnC_{34}H_{28}N_2O_{11}S_2Cl_2]$	840.38			7.77	48.54	3.33	3.33	7.61	20.94	8.44

			294-296	Purple	(7.74)	(48.50)	(3.30)	(3.29)	(7.58)	(20.90)	(8.40)
5	[$CdC_{34}H_{28}N_2O_{11}S_2Cl_2$]	888.02	>300 Dec	Purple	12.65	45.94	3.15	3.15	7.20	19.01	7.99
					(12.62)	(45.90)	(3.11)	3.10	7.15	19.00	7.95

FT-IR of ligand and Facilities

Table 2 lists the Schiff basis ligands' infrared spectrum data together with their complexes. For the aim of controlling coordination of sites, and that can be challenging in chelation, infrared spectra of complexes have been matched with those of free ligand. Transferrable to stretching shaking carbonyl group of the Schiff base ligand that has been elevated to a lower level of frequency in the infra-red spectra of complexes at 1168 cm^{-1}, the band in infrared spectrum of free ligands at 1172 cm^{-1} was obtained. The υ(M-O) confirms [7-10] hence the band appearance in (519-652) cm^{-1} range in infrared spectra of all complexes had occurred as consequence. In infrared spectra of facilities of Co (II), Nickel (II), Cd (II), and Zinc (II), fell to lower frequency that had been specified for harmonizing ligand with metal ion through N atom; the band at (1688cm^{-1}) in free ligand's infrared spectra could be the result of stretching vibration of imine group υ(C=N) changed to lower level of the frequency [8]. This was confirmed even more by presence of newfangled band in (518-652cm^{-1}) range that is transferable to υ(M-N) [11-15]. It has been shown that for all of the facilities the ligand had behaved as bi-dentate ligand that had been harmonized to metal ions by using N atom of imine group and 0 atom of naphthol group .

Table2: Properties of infra-red bands of ligand (L) absorption in addition to its complexes in cm-1

Compound	**υOH**	**υ (C=N)**	**υ (C-O)**	**υ (M-N)**	**υ (M-O)**
$C_{17}H_{11}NO_4SCl$	3225	1688	1150	--	--
$CoC_{34}H_{26}N_2O_{10}S_2Cl_2$	3238	1653	1166	652	519
$NiC_{34}H_{26}N_2O_{10}S_2Cl_2$	3228	1654	1163	650	591
$ZnC_{34}H_{26}N_2O_{10}S_2Cl_2$	3234	1658	1166	518	652

$CdC_{34}H_{26}N_2O_{10}S_2Cl_2$	3185	1686	1158	520	594

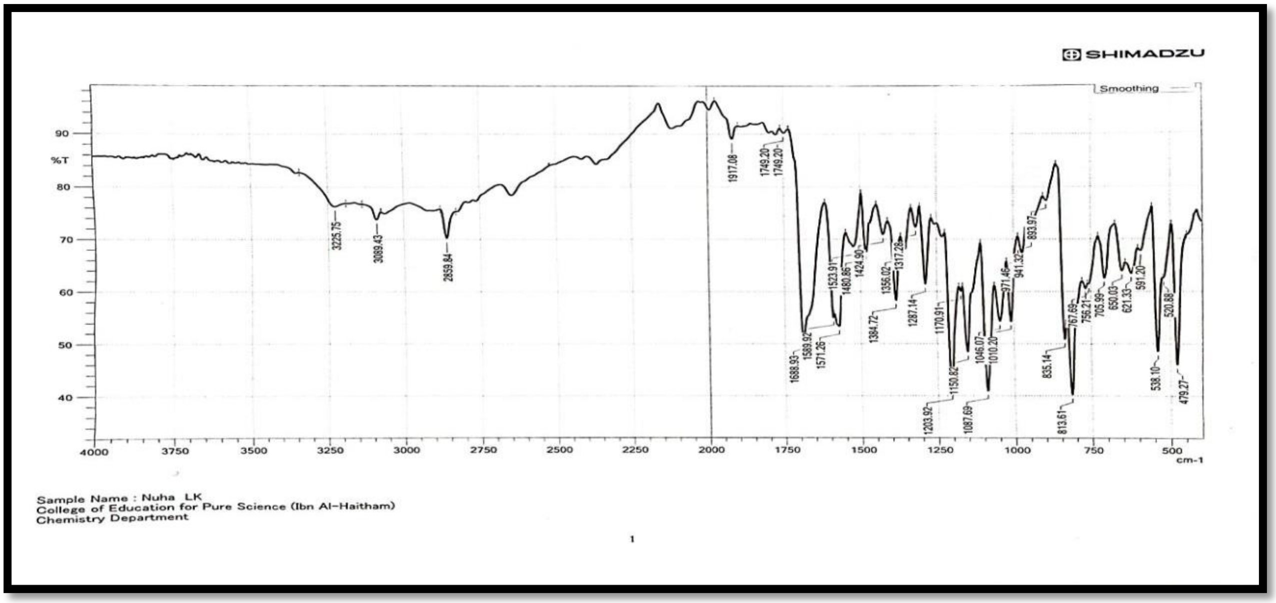


Figure (1) Infra-red spectra of ligand (L)

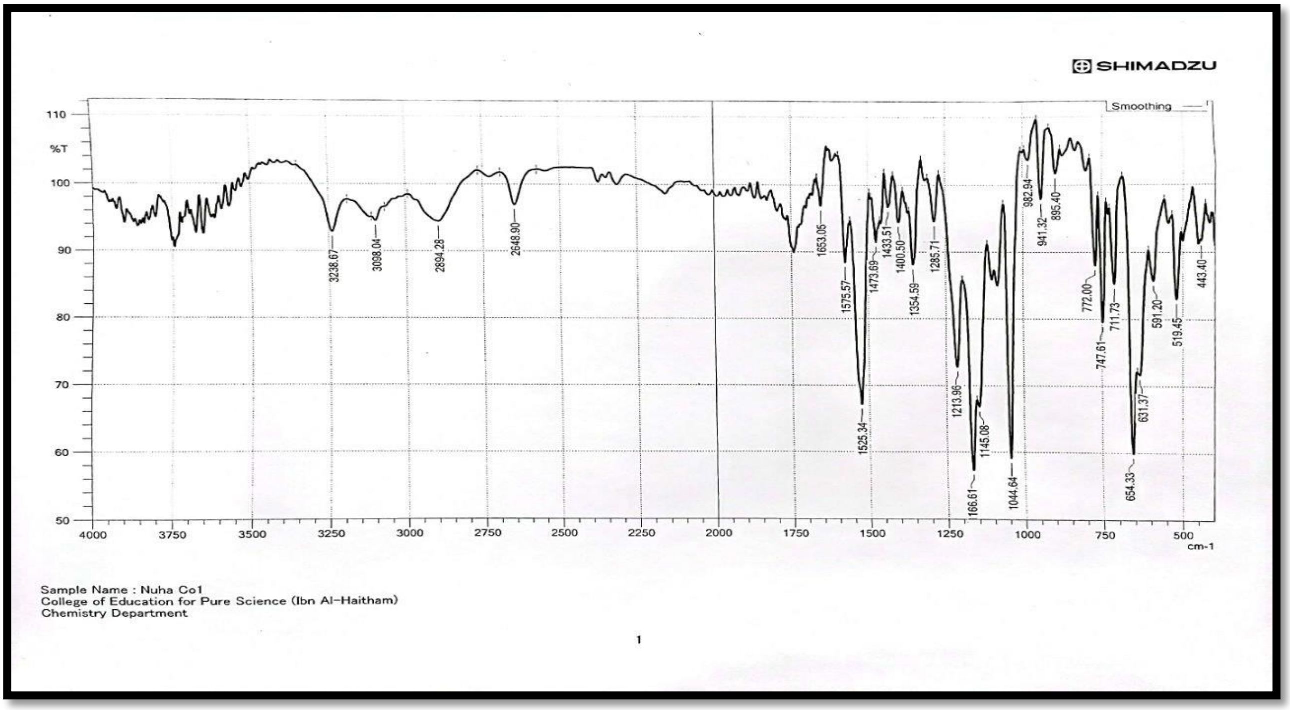


Figure (2) Infra-red spectra of complex $[Co(L)_2(H_2O)_2].H_2O$

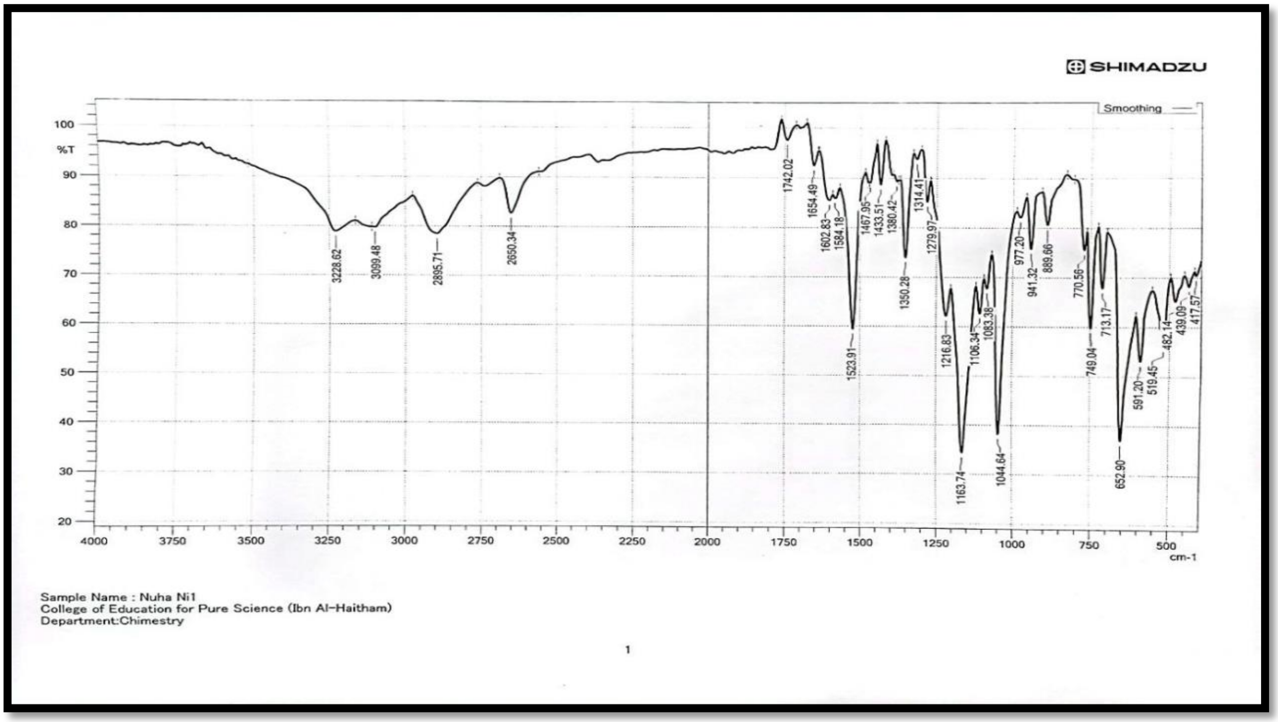


Figure3. Infra-red spectra of complex $[Ni(L)_2(H_2O)_2].H_2O$

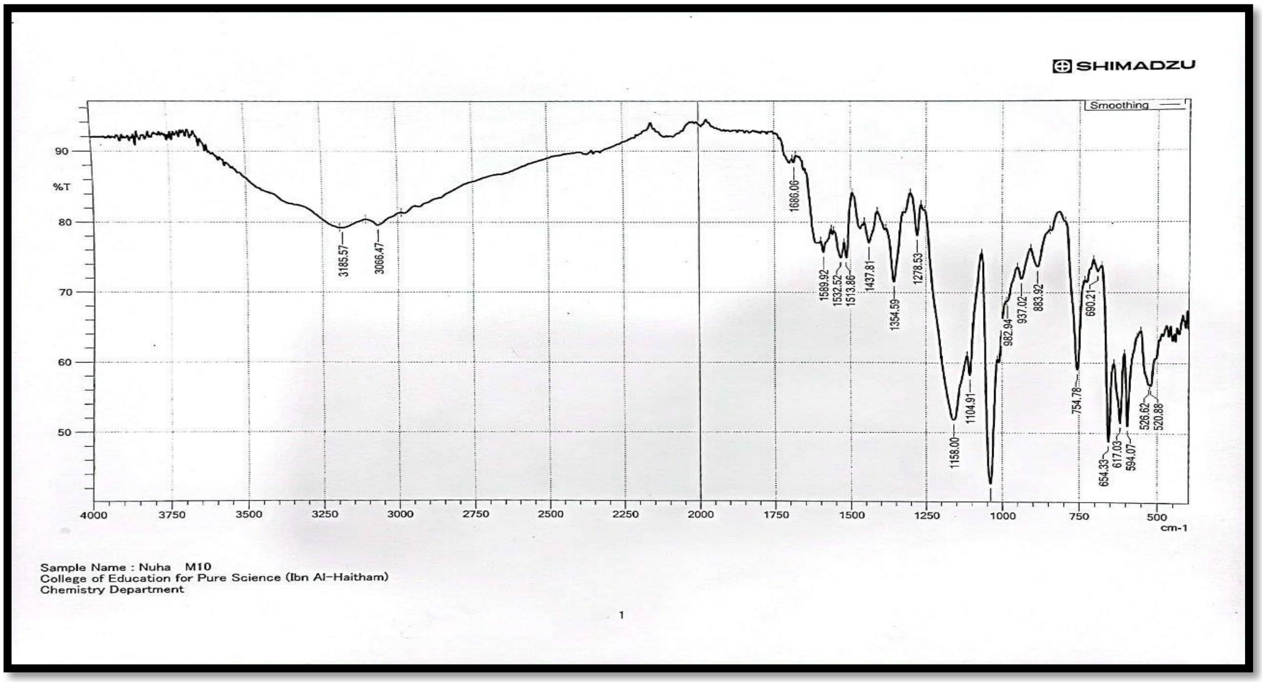


Figure4 Infra-red spectra of complex $[Cd(L)_2(H_2O)_2].H_2O$

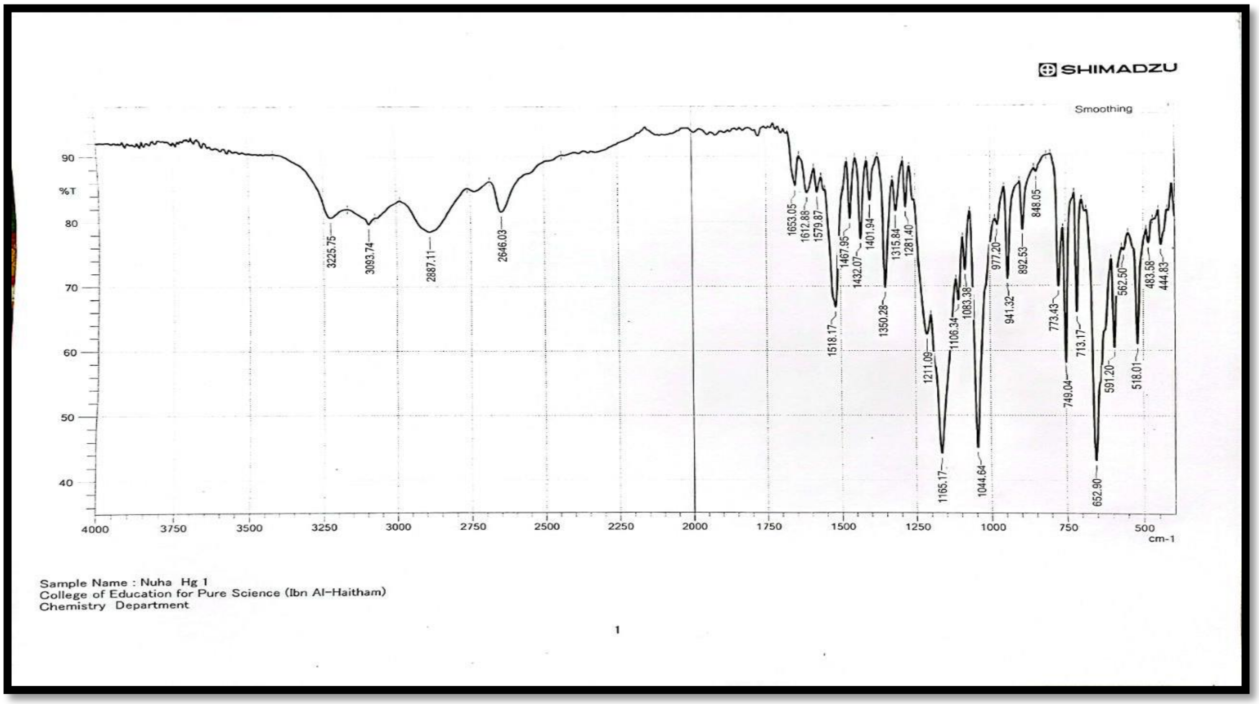


Figure5. Infra-red spectra of complex $[Zn(L)_2(H_2O)_2].H_2O$

UV-Vis

Deliberate UV-Vis of ligand and its Co (II), Ni (II), Cd (II) and Zn (II) complexes; spectra 1 data have been registered as shown in Table 3. Mostly 3 absorption top at (235nm) assigned to (π→π*), at (345nm) allocated to (n→π*, and at (470 nm) that had been allocated to Charge transfer [16-18] was defined as the UV-Vis of Schiff base ligands. In electronic spectra of all the designated sites, these electronic transitions had been raised either near higher or lower frequency levels [19-25].

Table 3: UV-Visible spectra of the ligand and it chelates complexes

Compounds	λ (nm)	$\upsilon(cm^{-1})$	max ε cm L/mol	Transition	$\Lambda mS.cm^2.mole^{-1}$
$C_{17}H_{11}NO_4SCl$	320 267	31250 37453	3890 3950	n→π* π→π*	
$CoC_{34}H_{26}N_2O_{10}S_2Cl_2$	234 275 338 543 927	427335 36363 29585 18416 10787	1630 660 490 390 4	Intra-ligand Intra-ligand $^4T_1g_{(F)}\rightarrow{}^4T_1\,g_{(p)}$ $^4T_1g_{(F)}\rightarrow{}^4T_1\,g_{(p)}$ $^4T_1g_{(F)}\rightarrow{}^4T_1\,g_{(p)}$	Octahedral
$NiC_{34}H_{26}N_2O_{10}S_2Cl_2$	215 257 359	46511 38910 27855	3350 3390 130	Intra-ligand Intra-ligand $^4T_1g_{(F)}\rightarrow{}^4T_1\,g_{(p)}$	Octahedral

	979	10214	30	$^4T_1g_{(F)}\rightarrow{}^4T_1\,g_{(p)}$	
$ZnC_{34}H_{26}N_2O_{10}S_2Cl_2$	229 272 325 396	43668 36764 30769 25252	1200 450 220 150	Intra-ligand Intra-ligand Intra-ligand $^4T_1g_{(F)}\rightarrow{}^4T_1g_{(p)}$	Octahedral
$CdC_{34}H_{26}N_2O_{10}S_2Cl_2$	234 497	42735 20120	1890 160	Intra-ligand $^3A_2\,g_{(F)}\rightarrow{}^3T_1g_{(P)}$	Octahedral

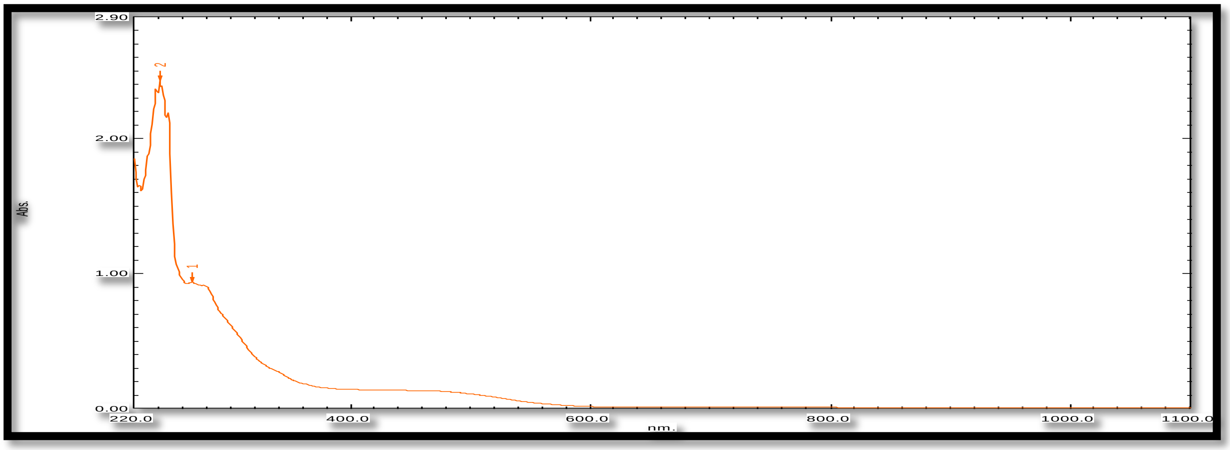


Figure (6) UV-Vis spectrum of the Ligand (L)

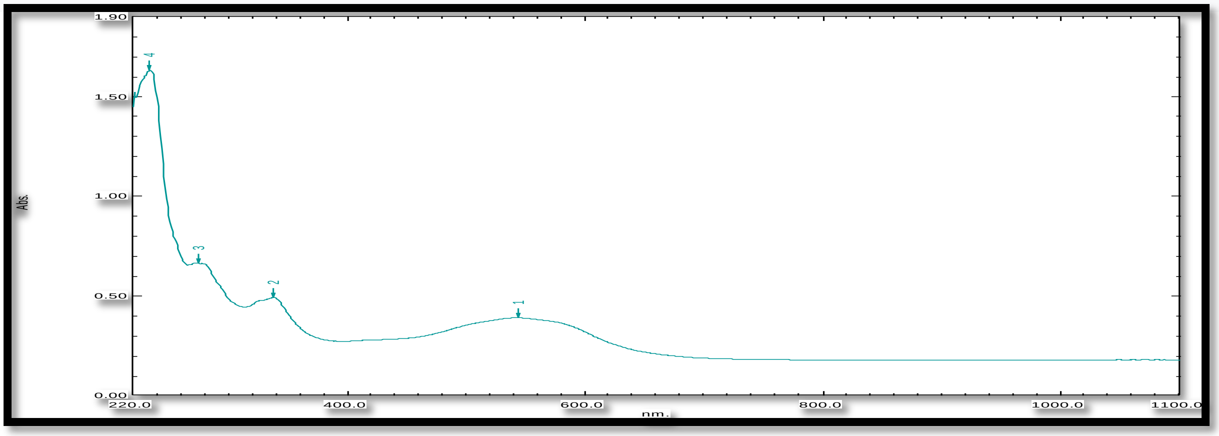


Figure7 UV-Visible spectra of the complex $[Co(L)_2(H_2O)_2].H_2O$

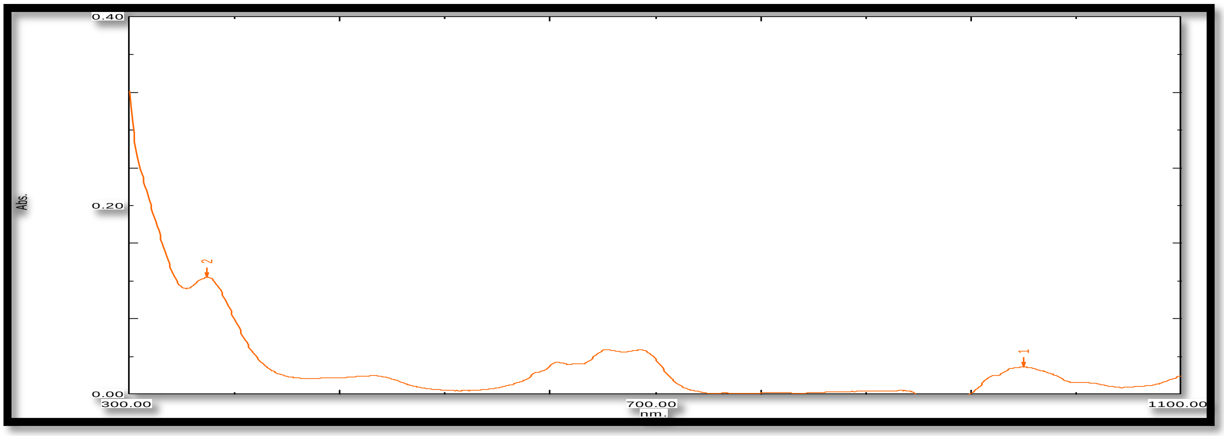


Figure (8) UV-Vis spectrum of complex $[Ni(L)_2(H_2O)_2].H_2O$

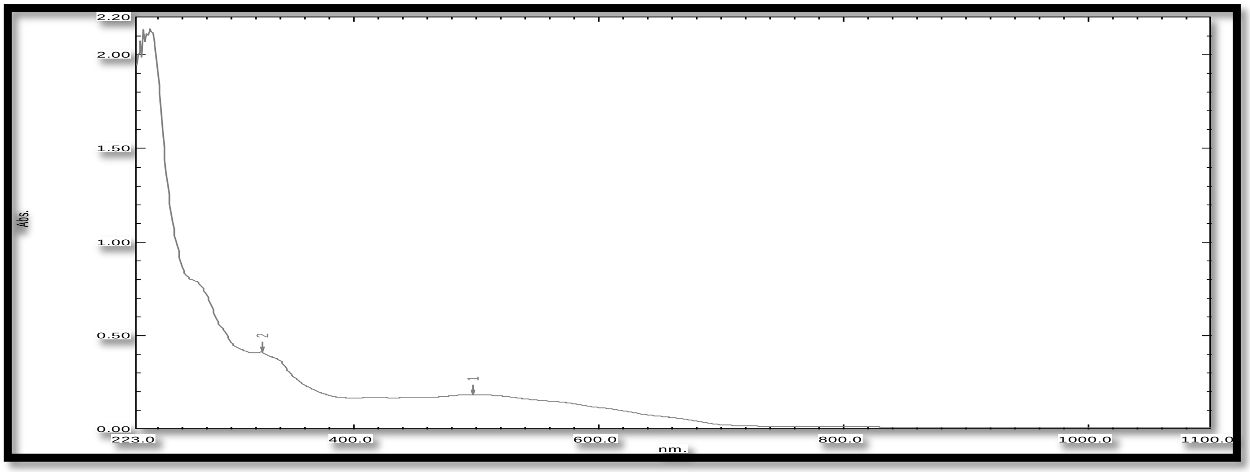


Figure8. UV-Visible spectra of the complex $[Cd(L)_2(H_2O)_2].H_2O$

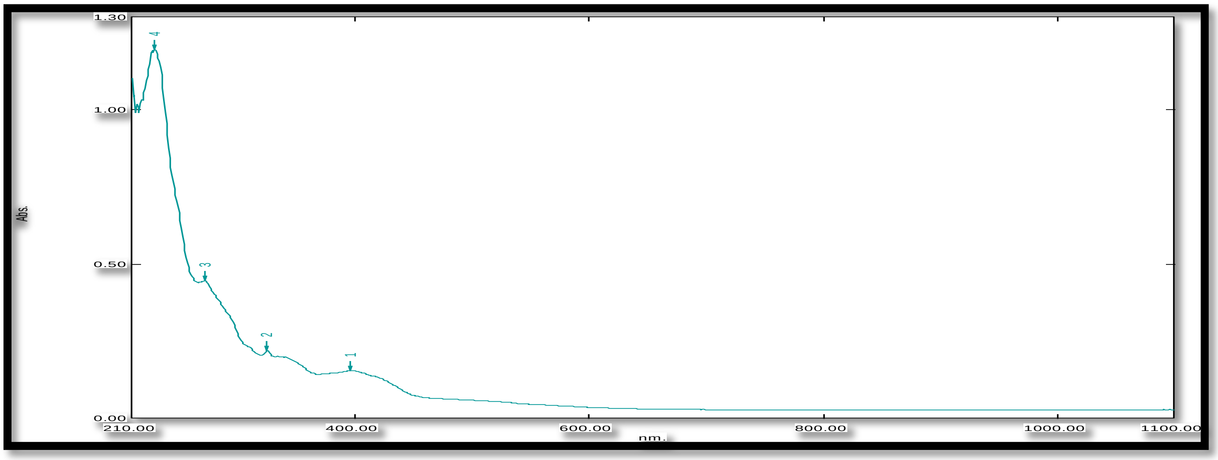


Figure (9) UV-Vis spectra of complex $[Zn(L)_2(H_2O)_2].H_2O]$

Conductivity and Magnetic measurement

Molarity measurement of complex solutions by preparing solutions with a concentration of 10^{-3} molar conductor in ethanol solvent at 298K and provided in Table4, and the prepared complexes were non-electrolytic (neutral) [26].

Table 4: Molar conductivity (Λm) of solutions of complexes in ethanol at a molar concentration of (10^{-3}) and magnetic moment values in B.M.

No.	Complex	μ_{eff} (B.M)	Conductivity | µS/cm	Ratio
1.	$[Co(L)_2(H_2O)_2].H_2O$	**4.82**	**9.8**	Neutral
2.	$[Ni(L)_2(H_2O)_2].H_2O$	**2.80**	**7.40**	Neutral
3.	$[Zn(L)_2(H_2O)_2].H_2O$	**0.00**	**12.0**	Neutral
4.	$[Cd(L)_2(H_2O)_2].H_2O$	**0.00**	**5.1**	Neutral

^{1}HNMR

In mix, as in the solid state, this phenomenon has been set by NMR and infrared spectra. 1HNMR ligand spectra in DMSO-d6 had revealed signal at the chemical shifting (δH =9.79ppm, s) credited to the one hydroxyl group phenol proton δ(OH) [27-28], and at (8.75ppm, s) credited to hydroxyl group sulfonic δ(OH) proton), and at (7.83ppm, s) credited to proton of. The values of the Resonance at chemical shifting (δH = 6.70 -7.6ppm, m) are flexible to aromatic ring protons and at (2.5 ppm, s) official of (DMSO).

Table5: 1HNMR spectrum data in DMSO-d6 solvent for the ligand SchL.

Ligand	Funct.Group	(ppm)□
L	(OH) for phenolic group	9.79) (1H,S)(
	(OH) for sulfonic group	8.75) (1H,S)(
	N=C-H	7.830) (1H,S)(
	Ar-H	6.7-7.6)(7H,m)(
	OCH$_3$) group(	3.0,3.2)(9H,S)(
	DMSO	2.59)(

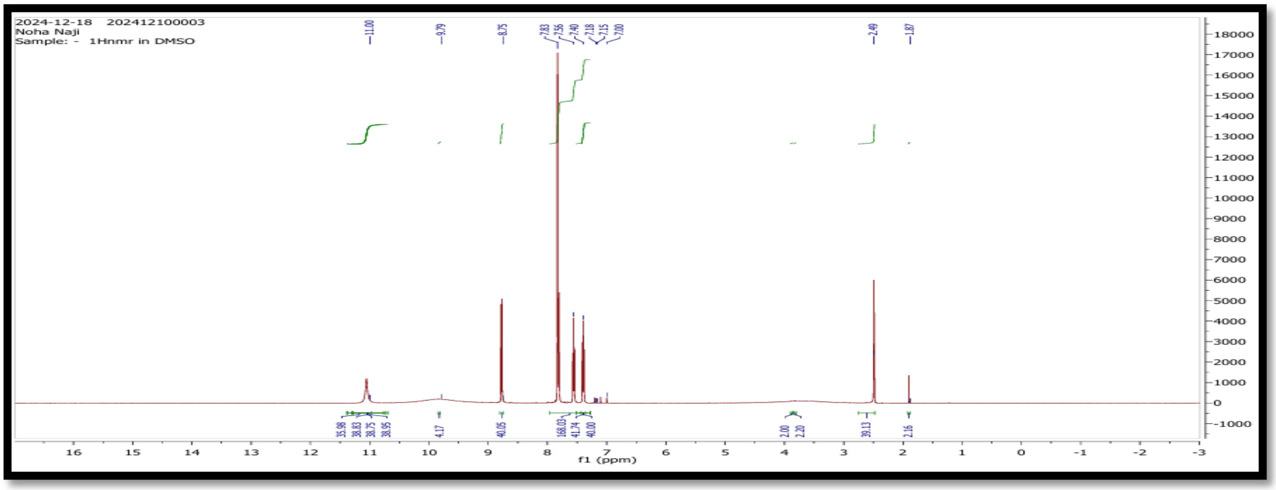


Figure (10) 1HNMR spectrum of Schiff ligand L

GC-mass of ligand and complexes

Other rubbles and their relative abundance and disintegration pattern have been provided together with an electrospray mass spectra of ligand displaying parent ion top at (M/Z) = (361.8). Parent ion top at (M/Z) = (841) is shown by electro-spray mass spectra of Zn (II), which also agrees with [M+], other fragments in addition to their relative abundance and disintegration configurations. Confirming parent ion top at (M/Z) = (888), which corresponds to [M+], further portions and their relative patterns of disintegration and abundance has been shown by electro-spray mass spectra of Cd (II). Thermal decomposition regarding Complexes [29,30] Co (II) and Ni (II). With a mean temperature degree of 10^o C/min and a 2.82mg weight, thermal research of the complex [$CoC_{34}H_{28}N_2O_{11}S_2Cl_2$] was conducted in an inert atmosphere of argon. The TGA curve exhibits the complicated four-stage weight loss process. Using an inert atmosphere of argon at a mean temperature degree of 10^{o}C/min, thermal research of complex [$NiC_{34}H_{28}N_2O_{11}S_2Cl_2$] was conducted with weight of (7.732mg) [31-32]. According to TGA curve, complex had one weight reduction phase.

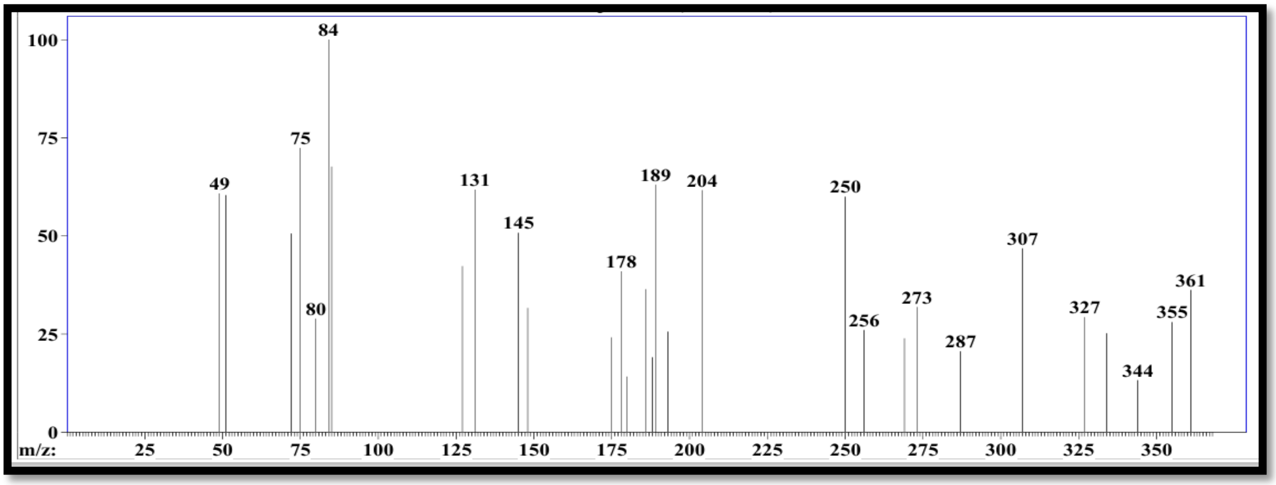
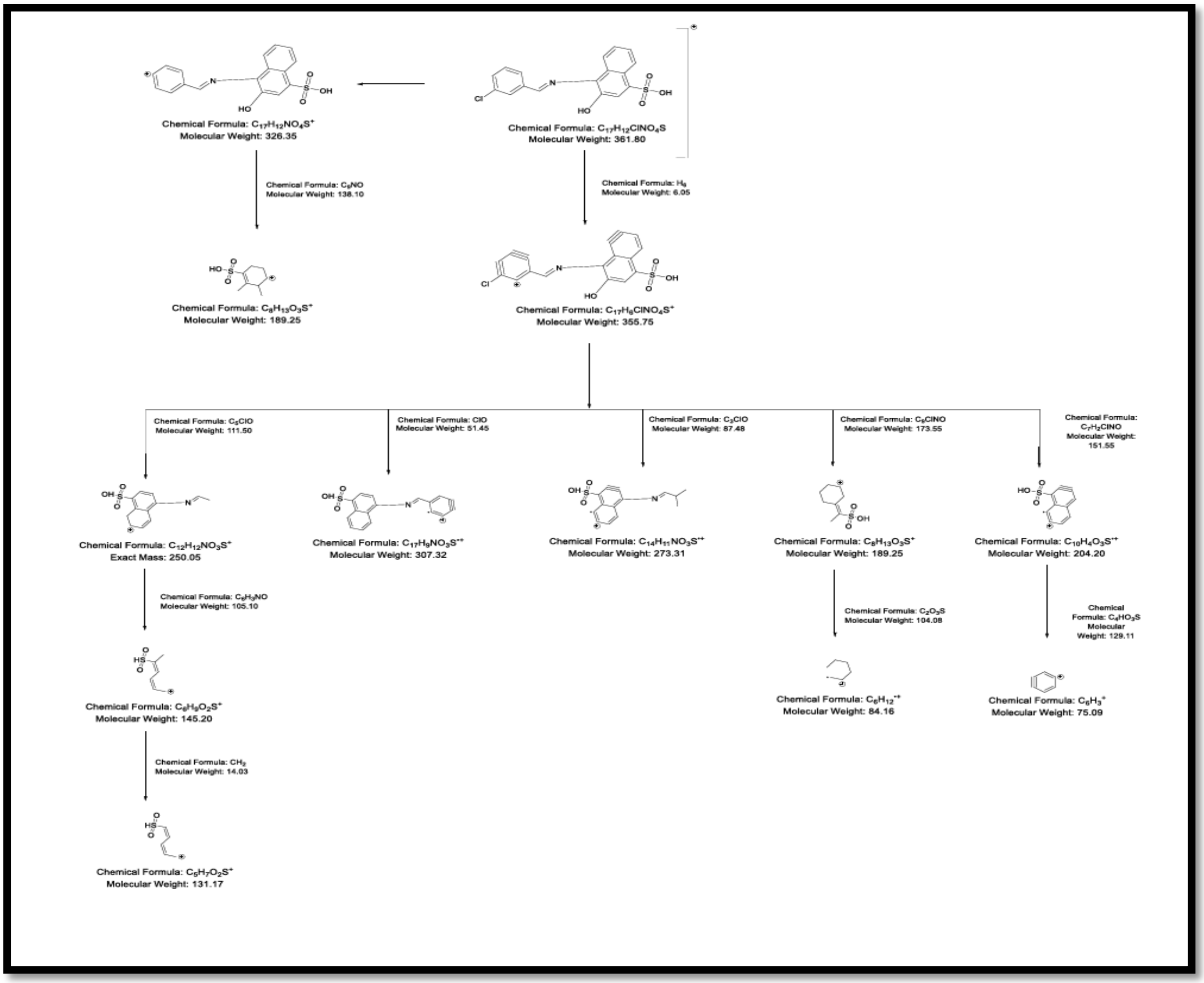


Figure (11) Mass spectrum of Schiff base ligand (L)



Scheme1 Suggested pathways for fragmentation of Schiff base ligand (L)

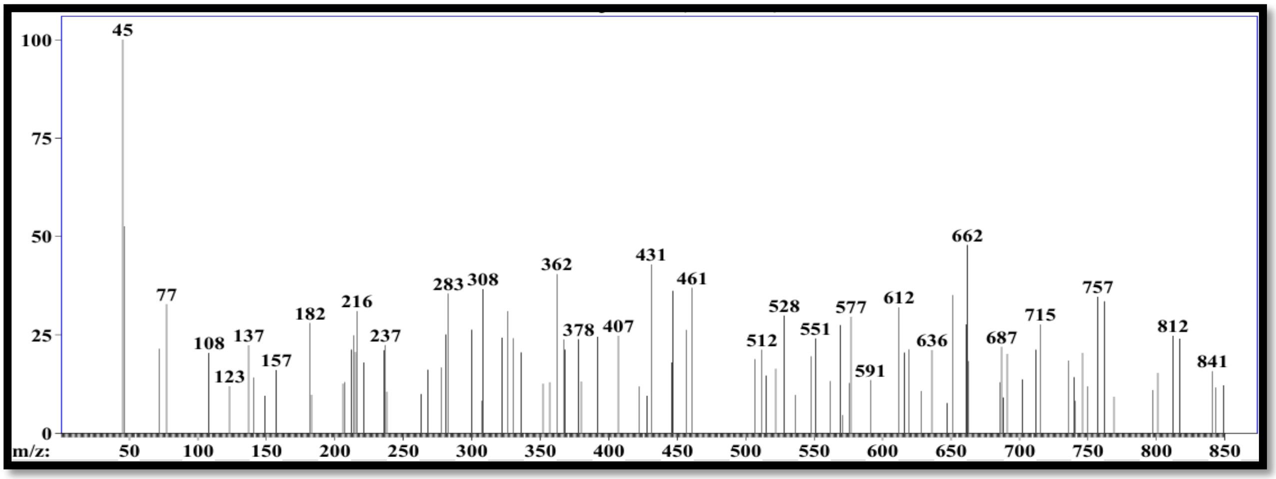
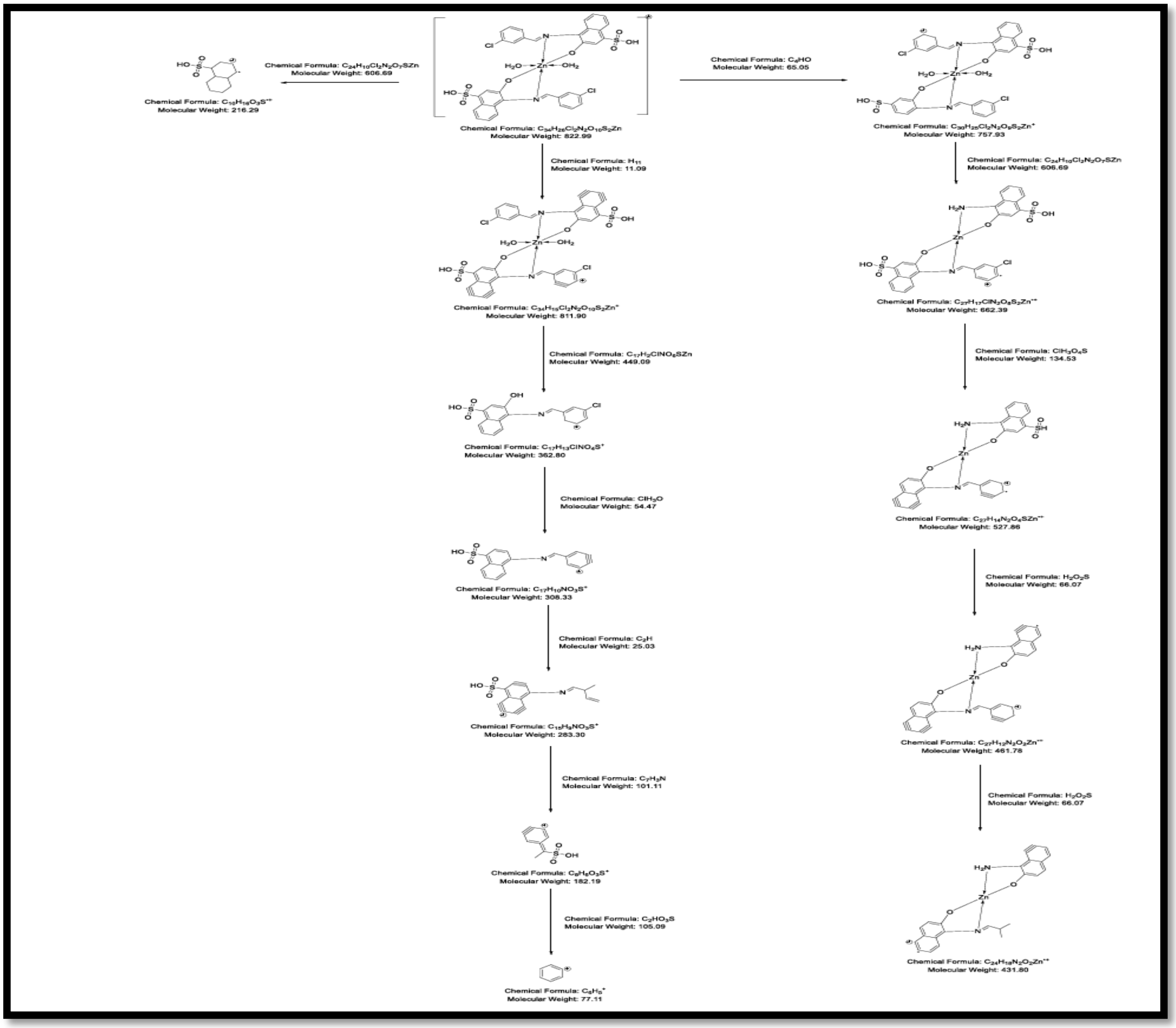


Figure12. Mass spectra of complex $[Zn(L)_2(H_2O)_2].H_2O$



Scheme (2) Proposed pathways for the fragmentation of complex $[Zn(L)_2(H_2O)_2].H_2O$

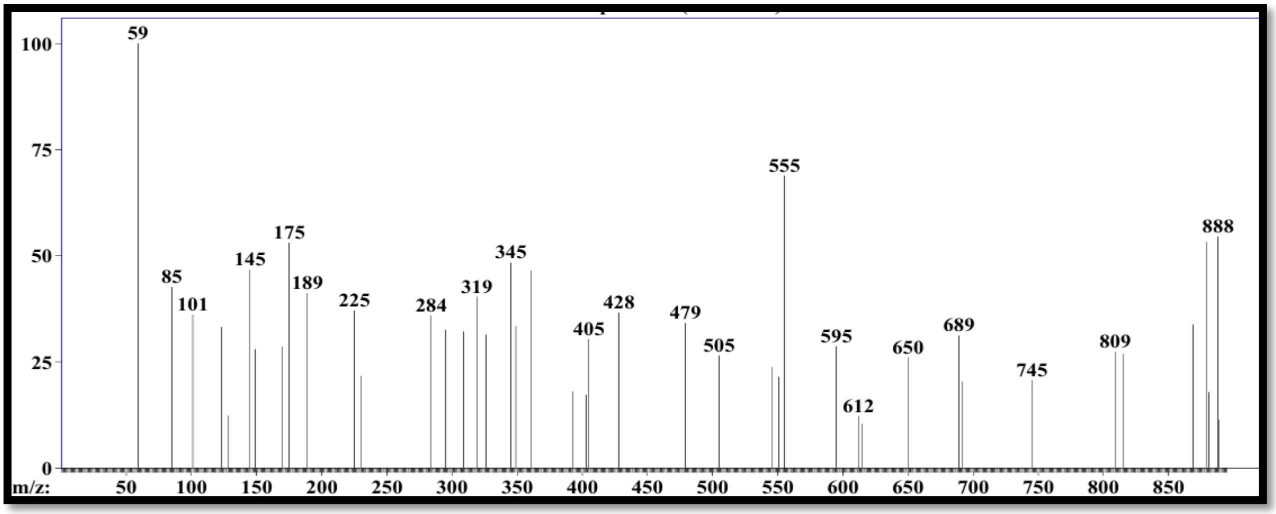
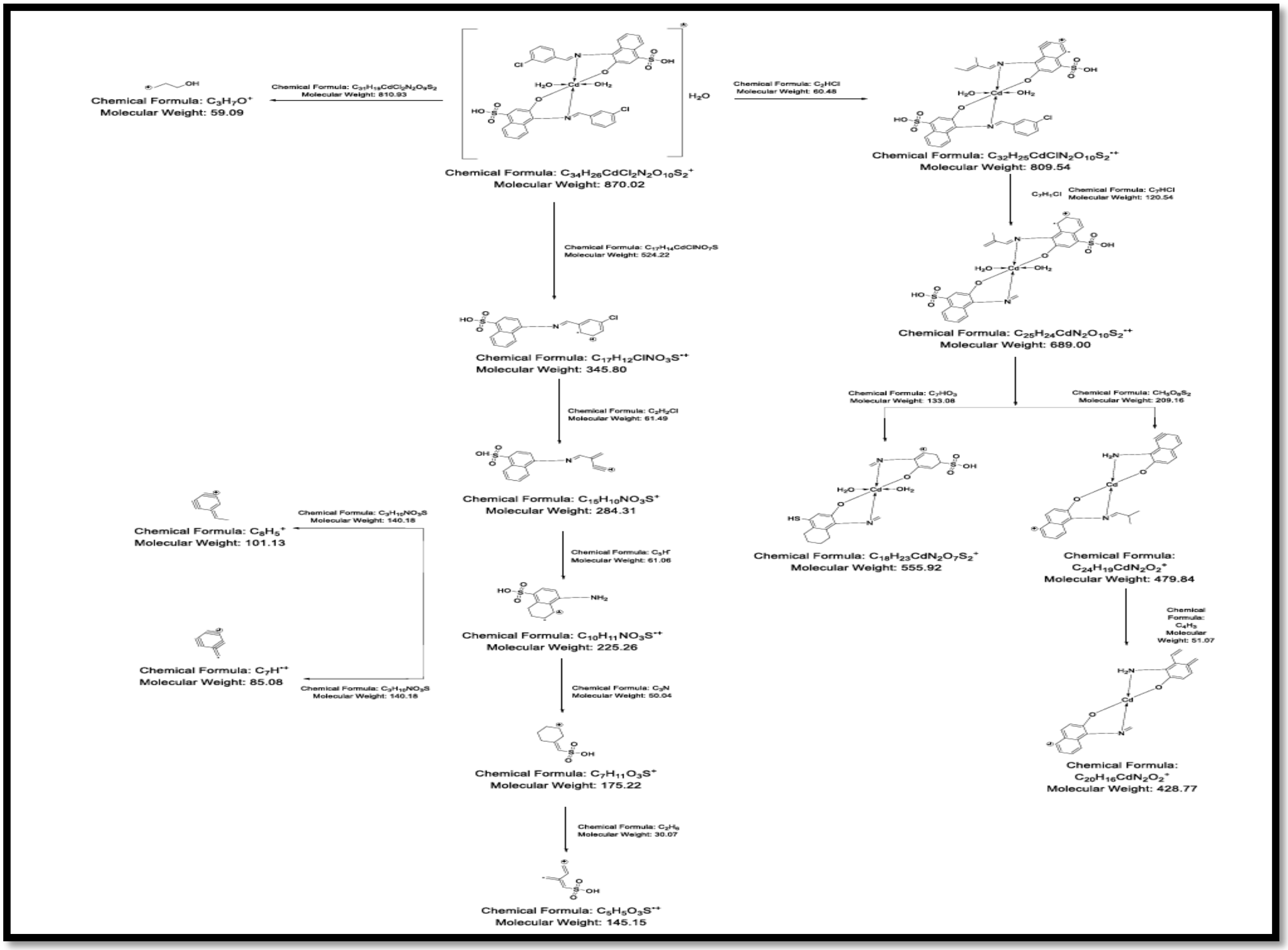


Figure (13) Mass spectra of the complex $[Cd(L)_2(H_2O)_2].H_2O$



Scheme (2) Proposed pathways for the fragmentation of the complex $[Cd(L)_2(H_2O)_2].H_2O$

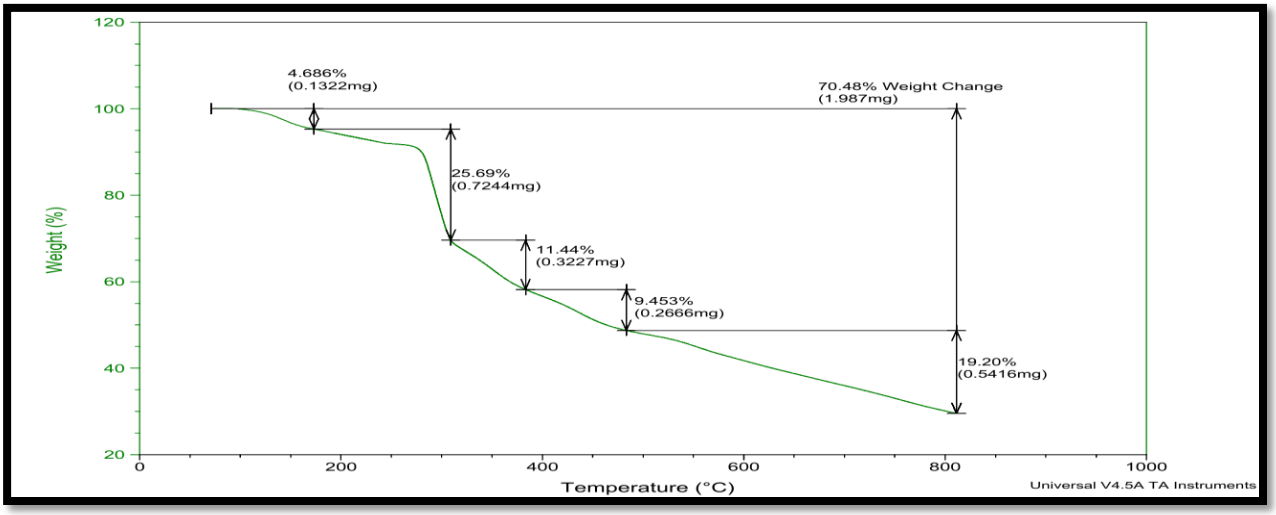


Figure14. Thermal dissociation spectra of complex [Co $(L)_2(H_2O)_2].H_2O$

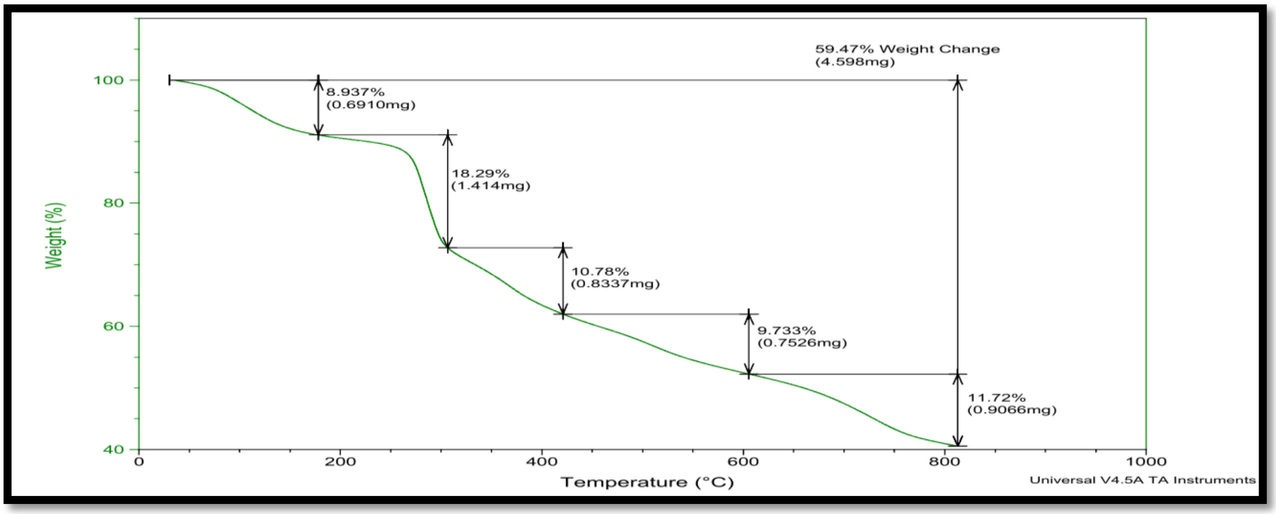


Figure15. Thermal dissociation spectrum of complex $[Ni(L)_2(H_2O)_2].H_2O$

Compound	Stage	Decomposition Temp. Initial-inal(℃)	Estimated(calculated)		Assignment
			Mass Loss	Total mass loss	
$[Co(L)_2(H_2O)_2].H_2O$	1	75-170	0.12(0.13)	0.862 (0.863)	$-2H_2O$ $-H_2$
	2	170-300	0.71(0.72)		-C6H6 -H2SO3 H_3O-
	3	300-370	0.30(0.31)		$-S0_2$ -HCN
	4	370-495	0.251(0.253)		$-C_3H_4Cl$
	5	495-810	0.531(0.533)		$-C_{12}HN$
$[Ni(L)_2(H_2O)_2].H_2O$	1	30-150	0.683(0.687)	3.157 (3.161)	$-4H_2O$ $-H_2$ $-C_7HNOCl$
	2	150-300	10384(1.389)		$-C_7HNOCl$
	3	300-410	0.821(0.824)		$-C_6H_3N$
	4	410-600	0.746(0.747)		-CHCl $-O_2$
	5	600-810	0.900(0.904)		$-SO_2$ $-H_2S$

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