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Synthesis, Spectral Identification, Thermal and Antioxidant Studies for Ni(II), Pd(II),Pt (IV) and Au(III) Complexes with New Azo Ligand Derivatives from Tryptamine

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Abstract

In this study, a new Azo ligand 5-((2-(1H-indol-2-yl)ethyl)diazinyl)-2-aminophenol is synthesized from a reaction of Tryptamine with 2-aminophenol. The ligand and their metal ion complexes Ni(II), Pd(II) , Pt(IV) and Au(III) have been synthesized and characterized by various analytical techniques, including elemental microanalysis, metal content, chloride-containing, measurement of electrical conductivity, magnetic susceptibility, ^{1}H and ^{13}C-NMR, FT-IR, UV-Vis, mass spectra (MS), and thermal analysis (TGA and DSC) curves. The DCS curve was used to calculate the thermodynamic parameters ΔH, ΔS, and ΔG. The characterization results promote the metal complexes of azo ligand structures. The results indicate that the ligand behaves as a trident N, N, and O donor ligand for each and every produced complex, and the molar conductivity of all complexes indicates that none of the complexes were electrolytes. The antioxidant activity of the synthesized compounds was evaluated and determined against the DPPH radical (1.1-diphenyl-2-picrylhydrazyl) and compared to that of a standard natural antioxidant, gallic acid. The results show that the metal complexes are more effective at scavenging free radicals.

Keywords: Gallic acid , Ligand , Thermal studies, Tryptamine and Mass spectrum.

1. Introduction

Tryptamine is one of the important chemical compounds in the medical and industrial fields due to its structure.These medications are ordinarily referred to as drugs. In addition, they are pharmaceutically and functionally diverse. The important auxiliary of the tryptamine class is its estimation of the neurotransmitter serotonin. Significant properties of tryptamine derivatives include antimicrobial, antifungal, antibacterial, antioxidant, anti-depressant, anti-inflammatory, migraine treatment, neuro transmitter and neuro modulator, vasoconstrictor and vasodilator, treatment for irritable bowel syndrome, diurnal rhythm regulation of many physiological systems, and nausea ,vomiting suppression from cancer chemotherapy and radiotherapy [1, 2]. The azo compound is the most significant and substantial class of synthetic dyes. According to the number of azo groups (N =N) attached to sp^2 hybridized carbon atoms, an azo molecule may have one or more of these groups [3–5]. These compounds play a general role in analytical, industrial, agricultural, and pharmaceutical applications and are widely employed in the textile, leather, printing, papermaking, drug, and food industries. A wide range of applications can be found in the chemistry of azo compound complexes. Because they contain atoms like oxygen, nitrogen, and sulfur, which enable them to link with various metal ions, these kinds of compounds are crucial in the biological sector [6–8]. Antioxidants are molecules that have the ability to neutralize oxidants and stop cell damage in the form of lipid, protein, and carbohydrate deterioration. It prevents the formation of free radicals. In the role of "free-radical-scavengers," antioxidants stop or delay the harm these free radicals cause. In our bodies, free radicals that are damaging to us are neutralized by antioxidants. It performs the role of a reducing agent, eliminating free radical intermediaries and halting further oxidation [9]. The various analytical techniques used to assess antioxidant capability can be divided into four groups: spectrometry, electrochemistry, chromatography, and fluorescence. Blois created this technique in 1958 to measure the antioxidant activity using a stable free radical,, -diphenyl-picrylhydrazyl(Mwt = 394.3 formula DPPH= $C_{18}H_{12}N_5O_6$) [10,11]. The assay is used to determine how well antioxidants can scavenge it[7].In this study, a novel Azo ligand is synthesized from a reaction of Tryptamine with 2-aminophenol and their metal complexes and characterized by various analytical techniques. The DPPH radical scavenging technique was used to assess the antioxidant capacities of metal complexes and the ligand.

2.Experimental

2.1 Materialsand Measurements

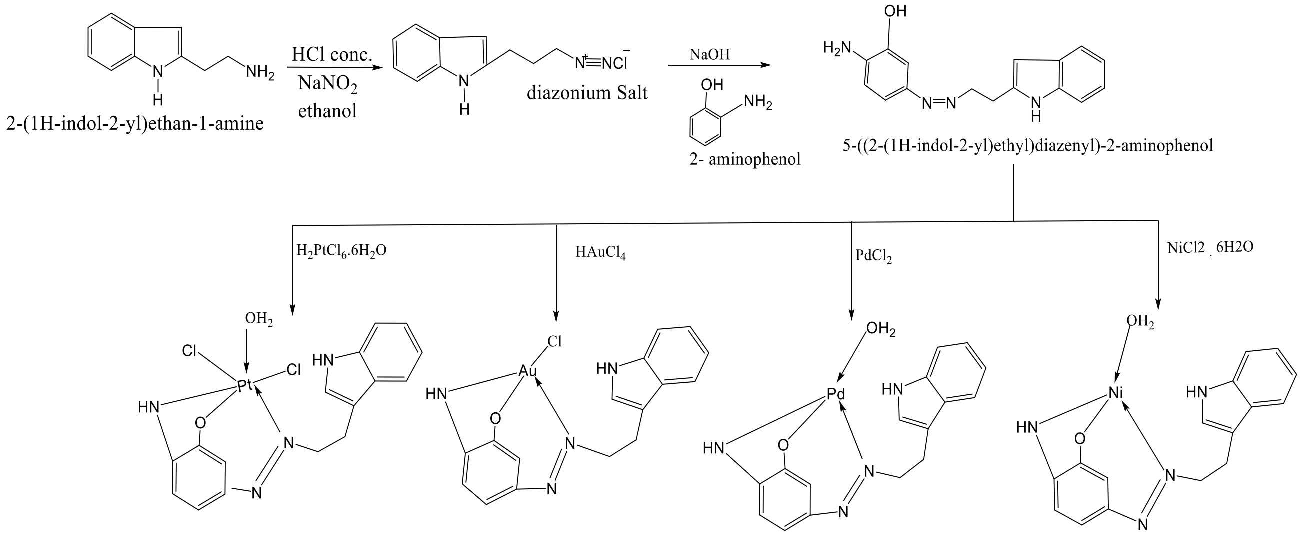
All chemicals were supplied commercially and utilized without further purification. All organic solvents were commercially available, distilled, and dried by appropriate methods. 1H and 13C NMR spectra were obtained from a Brucker (500MHz) spectrometer. UV-1800 Shimadzu spectrophotometer was used to record the UV-visible absorption spectra. Mass spectra were measured. On QP50A: DI Analysis ShimadzuQP-2010-Plus (E170Ev) spectrometer IR spectra were examined by IR Prestige-21. The Euro vector model EA/3000, single-V.3.O-single, was employed to obtain (C, H, and N) elemental analyses. Utilizing a Shimadzu (A.A) 680 G atomic clock, metals were identified. A conductivity meter (WTW) was used to detect conductivity while it was at room temperature with DMSO solutions. On the QP50A: DI Analysis ShimadzuQP-2010-Plus (E170Ev) spectrometer, electron impact (70 eV) mass spectra were captured. A gravimetric estimation of the chloride concentration was made. The balancing magnetic susceptibility model MSR-MKI was used to measure magnetic characteristics. Perkin-Elmer Pyris Diamond DS/TGA was used for all prior sorts of thermal analysis.

2.2.Synthesis of Azo dye ligand (L):

Tryptamine (1 g, 0.006 mol) has been dissolved in a mixture of 2 ml HCl and 15 ml of ethanol at 0–5 °C during refrigeration. Gradually add (10%, 0.43 g, 0.006mol) of $NaNO_2$ to avoid temperature increases of up to 5°C. After the reaction has been stirred for approximately 30 minutes, add the (0.671g, 0.006 mol) of 2-aminophenol dissolved in 10 ml of ethanol. Then, 3ml of a 1M NaOH solution was added, and the precipitation of a dark brown-colored azo ligand was observed. This was weighed after being filtered, collected, and given time to dry. Its melting point was 191 °C, and its yield was 81.5%.

2.3.Synthesis of metal ionscomplexes with ligand (L):

Complexes were prepared by adding a solution of 1 mmol of metal salts in 10 ml of ethanol dropwise to an ethanolic solution of 15 ml from 1 mmol Azo ligand. The resulting mixture was refluxed for 2 hours. The solid complexes were separated, and any unreacted components were removed by briefly immersing them in hot ethanol. After drying, gathering, and weighting the complexes, **Scheme 1** below shows how ligands (L) and their complexes are prepared.



Scheme 1: Preparationforligand (L) and theirmetal complexes

3. Result and discussion

The physical characteristics of the produced compounds, the elemental information of the C.H.N.O. studies, and the chloride and metal contents are listed in **Table 1**. It was evident from the experimental data that the theoretical value was supported. The proposed formula was confirmed. The molar conductance values of the soluble complexes with ligand (L) in DMSO solvent in 1×10^{-3} M solution at room temperature refer to the complexes of ligand (L). All complexes have a non-electrolytic nature.

Table 1. Analytical information and physical characteristics of the ligand Land its metal complexes

Comp. formula	**M.wt**	**%M (Expert) Calc**	**%Cl (Expert Calc**	**(Expert) Calc**				**color**	**m.p°C**	**Λm (S.cm^2.Mol^{-1})**
				%C	%H	%N	%O			
L **$C_{16}H_{16}N_4O$**	280.32	**-**	**-**	(67.89) 67.84	(5.74) 6.71	(21.01) 19.78	(7.00) 5,65	reddish brown	192-195	-
[Ni(L)(H_2O)] **$C_{16}H_{16}N_4NiO_2$**	35.69	(15.51) 16.54	-	(53.04) 54.13	(5.13) 4.51	(16.31) 15.79	(10.10) 9.02	green	d 224	14
[Pd(L)(H_2O)] **$C_{16}H_{16}N_4PdO_2$**	402.42	(27.72) 26.44	-	(46.64) 47.71	(4.83) 3.97	(14.32) 13.91	(7.62) 7.95	reddish brown	d 254-256	10
[Pt(L)(H_2O)Cl_2] **$C_{16}H_{16}N_4PtO_2Cl_2$**	561.98	(33.03) 34.71	(12.94) 12.71	(34.31) 34.16	(2.50) 2.84	(10.99) 9.96	(6.09) 5.69	pink	d212	7
[Au(L)Cl] **$C_{16}H_{14}AuClN_4O$**	510.41	(39.09) 38.58	(7.71) 7.50	(36.39) 37.61	(3.03) 2.74	(11.41) 10.97	(3.31) 3.13	purple	d202-205	16

Calc=calculated
d= decompose

3.1. (^{1}H and ^{13}C) NMR spectrum

The ^{1}H-NMR spectrum of ligand (L_2) was measured using TMS as an internal reference and DMSO-d_6 as the solvent, as illustrated in **Figure 1.** They are listed in **Table 2**. There have been many chemical changes discovered by the ^{13}C-NMR Spectra to the carbon atoms on the sites [(C1)196.1, (C2) 157.04, (C3) 134.21, (C4) 131.62, (C5)180.52, (C6)106.38, (C7) 24.02, (C8) 49.33, (C9)122.41, (C10) 162.11, (C11)146.11, (C12)151.87, (C13)145, (C14)115.34, (C15)166, and (C16) 152.16], respectively. **Figure 1** shows the ^{13}CNMR of the ligand (L) [11–14].

Table 2:^{1}H-NMR spectral data for ligand(L)

Chemical shift δ (ppm)	**Functional. Group**
1.61-1.94	((4H)t,CH_2-CH_2)
2.51-2.61	DMSO
6.76	((1H)s,CH-NH Indole)
6.94	((1H)s,CH-OH)
7.58-7.60	((1H) d,CH-NH_2).
7.83-7.86	((1H)d, CH-N=N)
7.96-8.04	((4H)m,CH arom)
9.01	((2H) s,NH_2)
10.52	((1H) s,OH).
11.30	((1H)s,N-H)

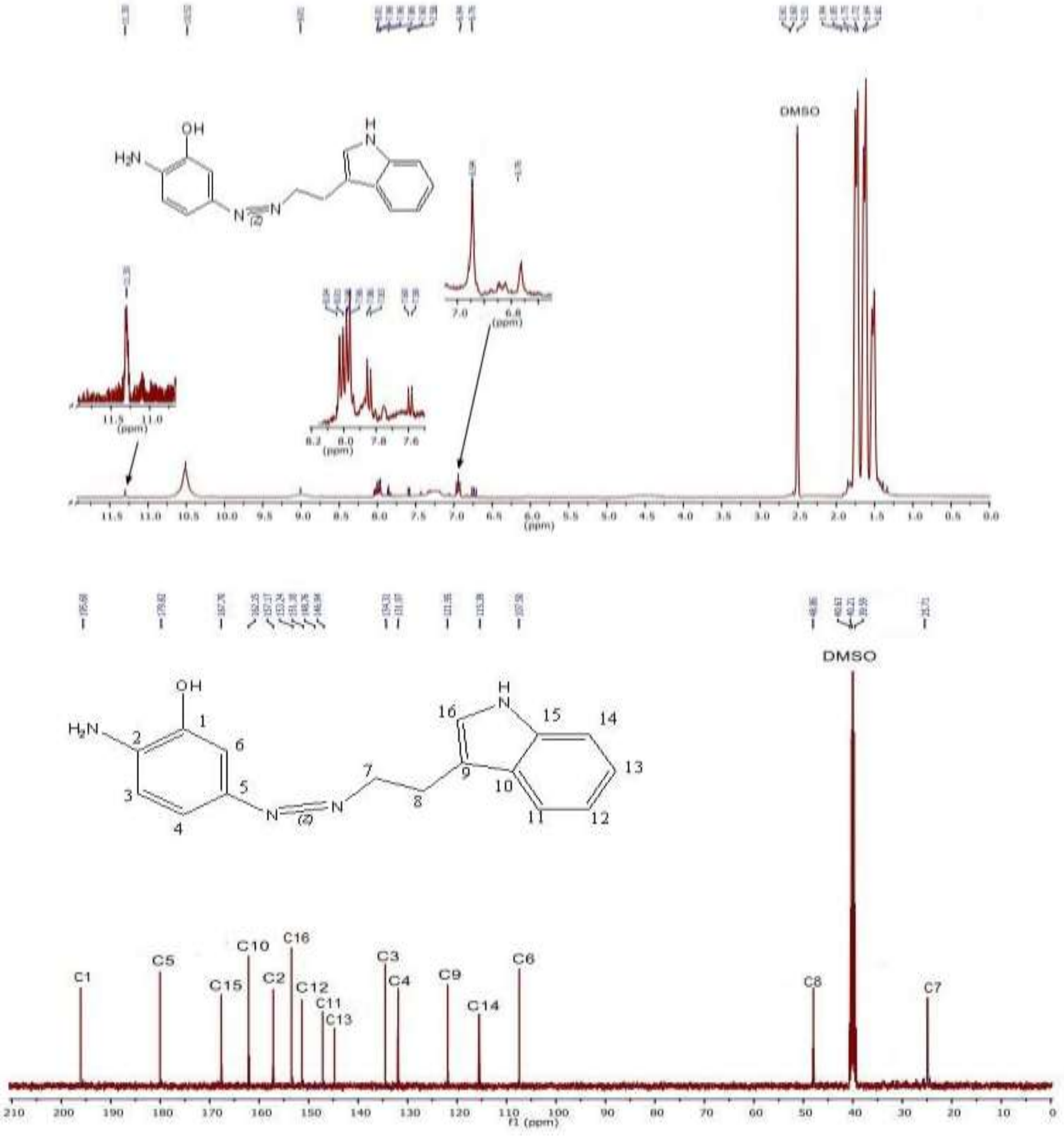


Figure 1.^{1}Hand ^{13}CNMR spectrum for ligand (L)

3.2 Mass spectrum of ligand and complexes

The mass spectra of ligands (L) and their complexes show a parent peak and a pattern of fragmentation ions that are easy to see. **Figure 2** displays the mass spectrum for ligand (L). The molecular ion peak, which corresponds to the ligand formula weight, has peaks at m/z=280.33.The spectrum exhibits other peaks at (m/z) (164.18, 116.14, 84.10,83.11,,67.11 and 56.09). The pattern of these peaks corresponds to C8H10N3O+, $C_8H_6N^+$,$C_4H_6NO^+$,$C_4H_7N_2^+$, $C_5H_7^+$and $C_3H_6N^+$). The mass spectral for Pd(II) complex, **Figure 3** the molecular ion peak, which corresponds to the ligand formula weight, has peaks at m/z=402.88 and shows many peaks at m/z = 384.73, 254.56, 139.45, 130.17, and 103.10. The pattern for these peaks may be assigned to various fragments with ($C_{16}H_{14}PdN_4O^+$, $C_7H_6PdN_3O^+$, C_9H_8N, H3PdNO+, C9H8N+,$C_9H_8N^+$and $C_6H_3N_4^+$) respectively. In **Figure 4**, the mass spectrum of Pt(IV), the molecular ion peak, which corresponds to the ligand formula weight, has peaks at m/z=562.33. And other peaks at (m/z) (413.39, 356.24, and 116.14) might be related to

($C_{16}H_{14}N_4OPt^+$, $C_8H_7N_3OPt^+$and C8H7N+), respectively. In the mass spectrum of the Au(III) complex, the molecular ion peak, which corresponds to the ligand formula weight, has peaks at m/z = 510. And other peaks at (m/z) (475.28, 345.11, 230, 130.17, and 103.10) might be related to ($C_{16}H_{14}N_4AuO^+$, $C_7H_6N_3AuO^+$, $C_9H_8N^+$ and $C_6H_3N_2^+$) respectively [15]. The pattern of fragmentation is summarized in **Scheme 2**.

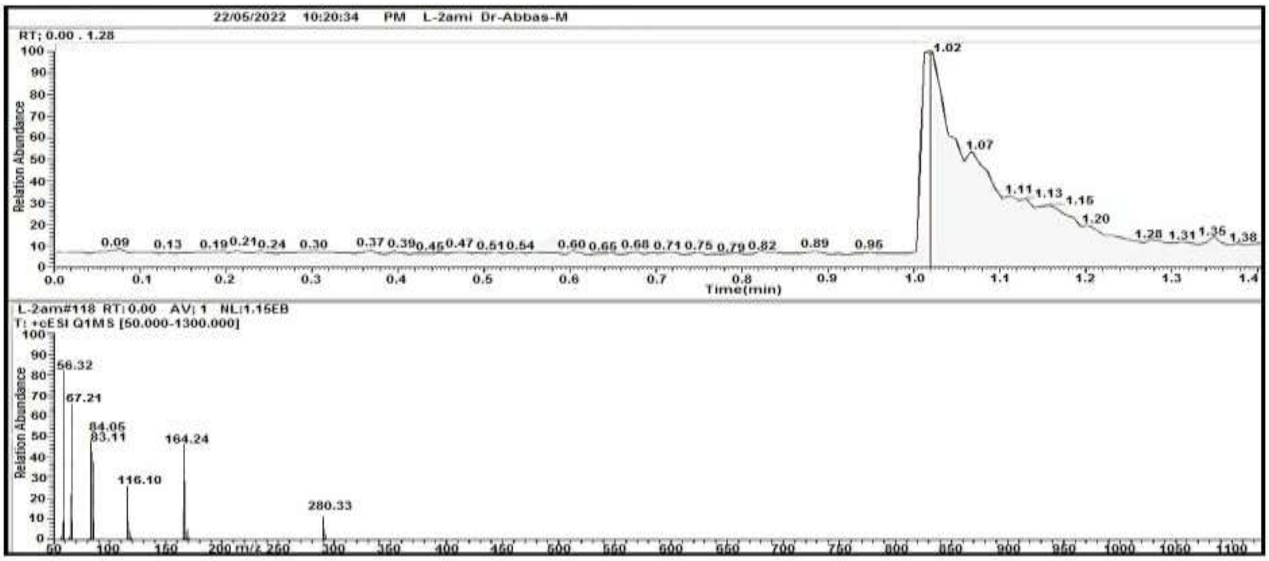


Figure 2. mass spectrum for ligand

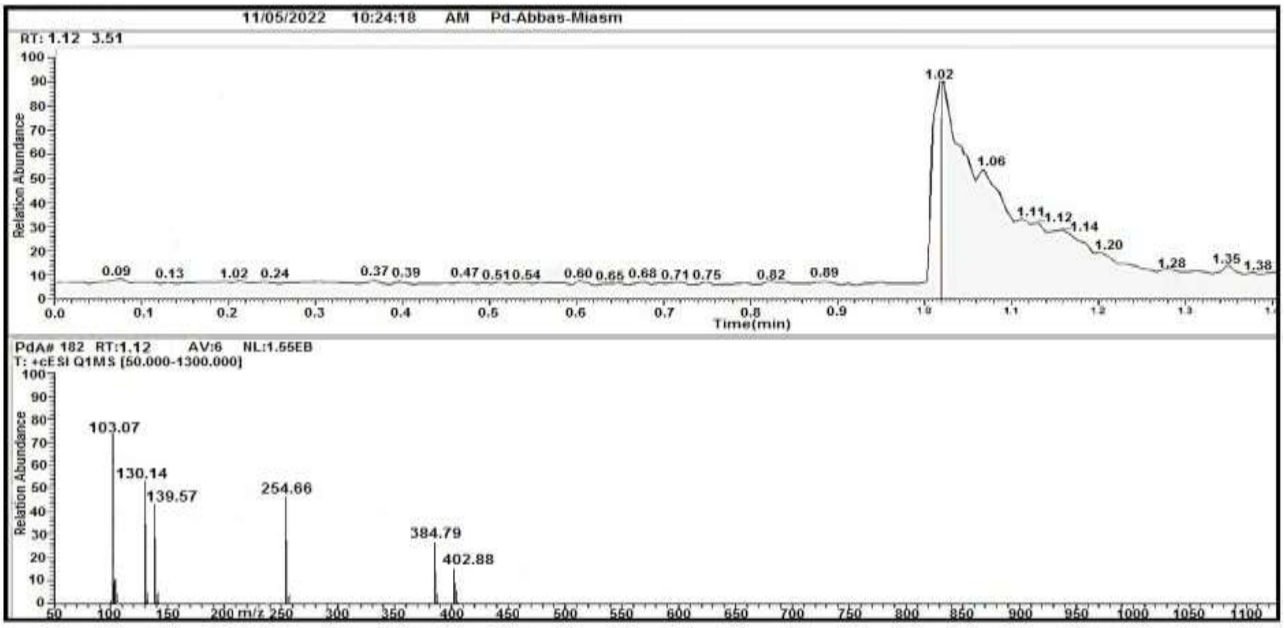


Figure 3. mass spectrum for Pd(II) complex

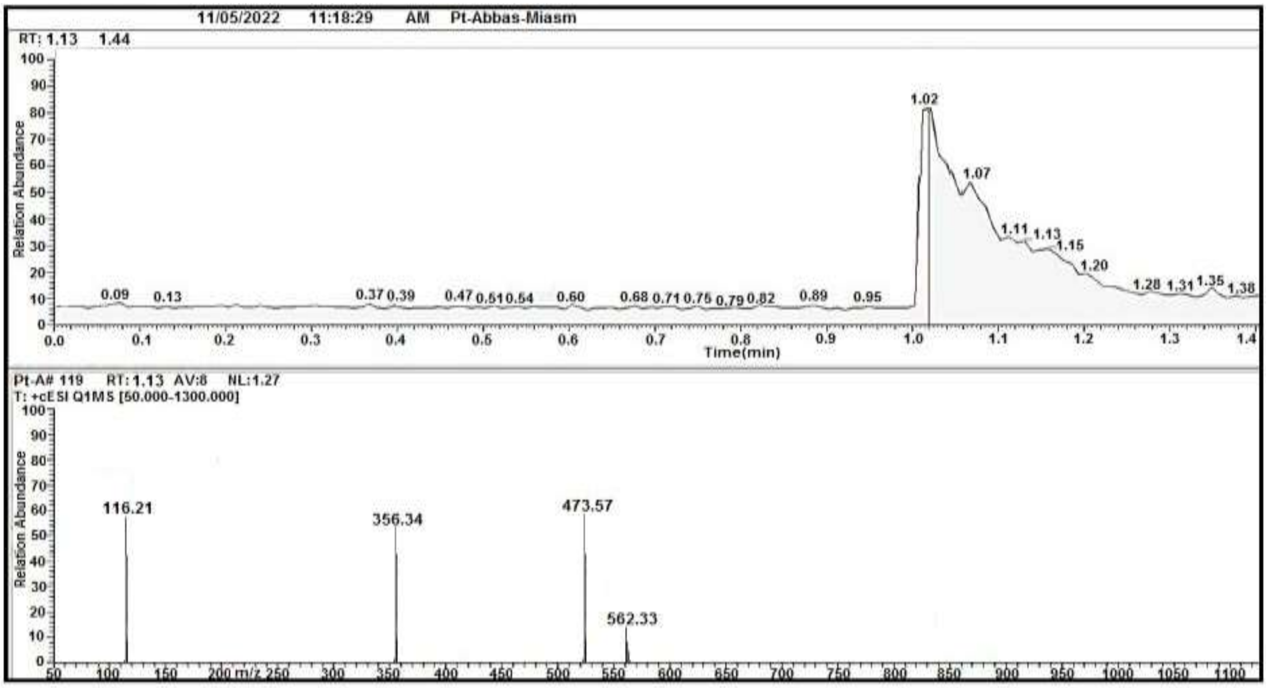
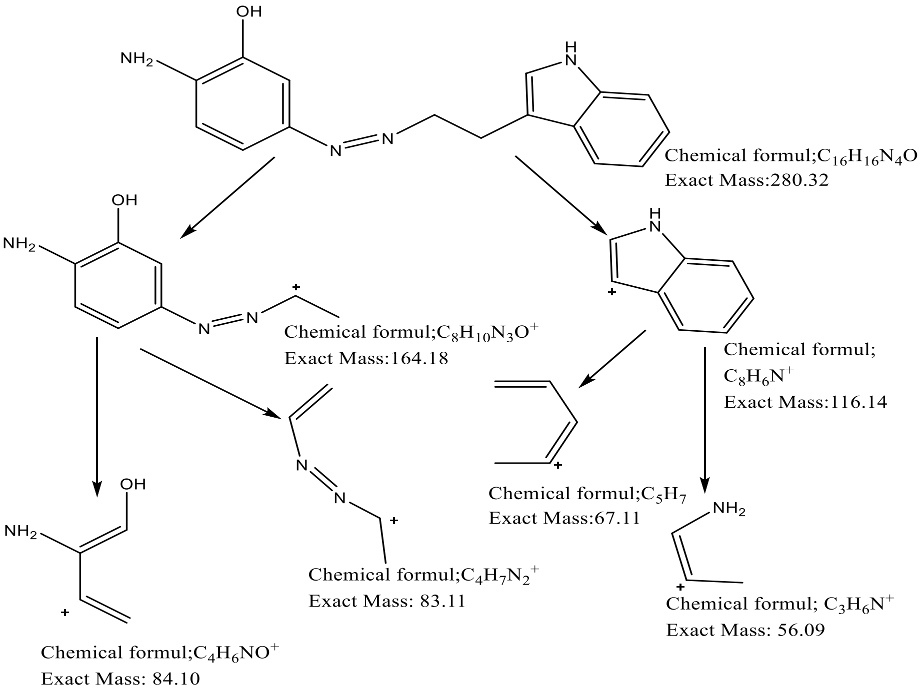
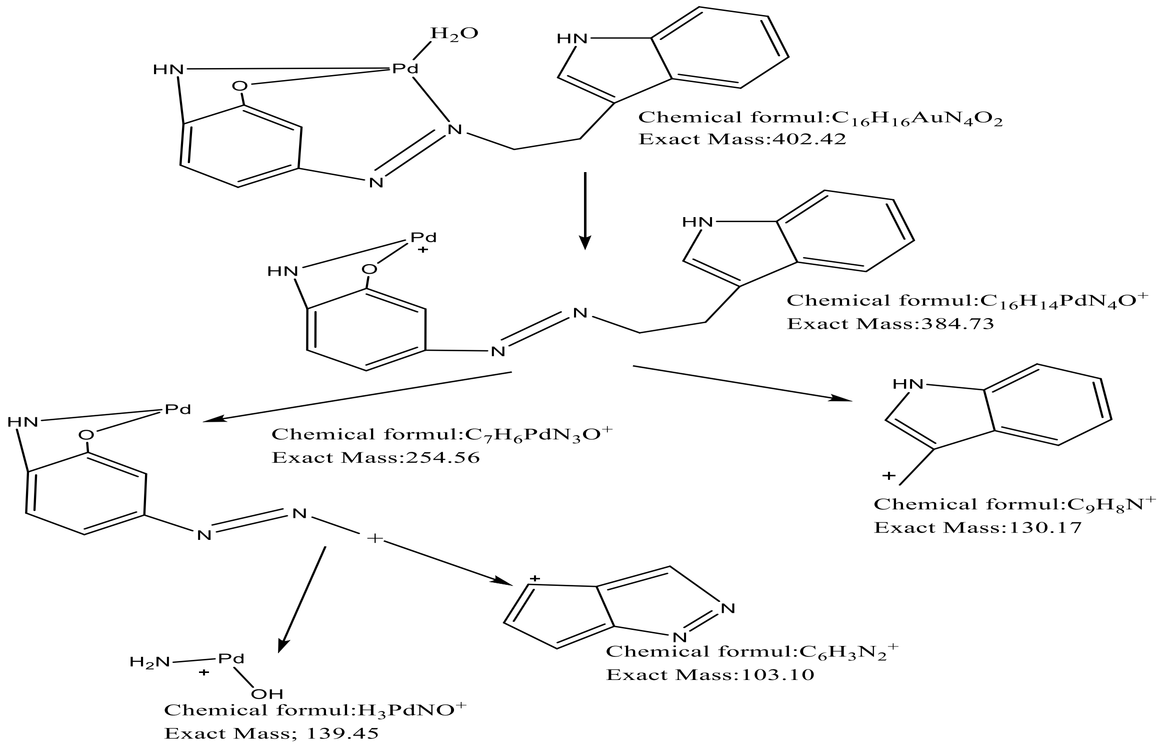
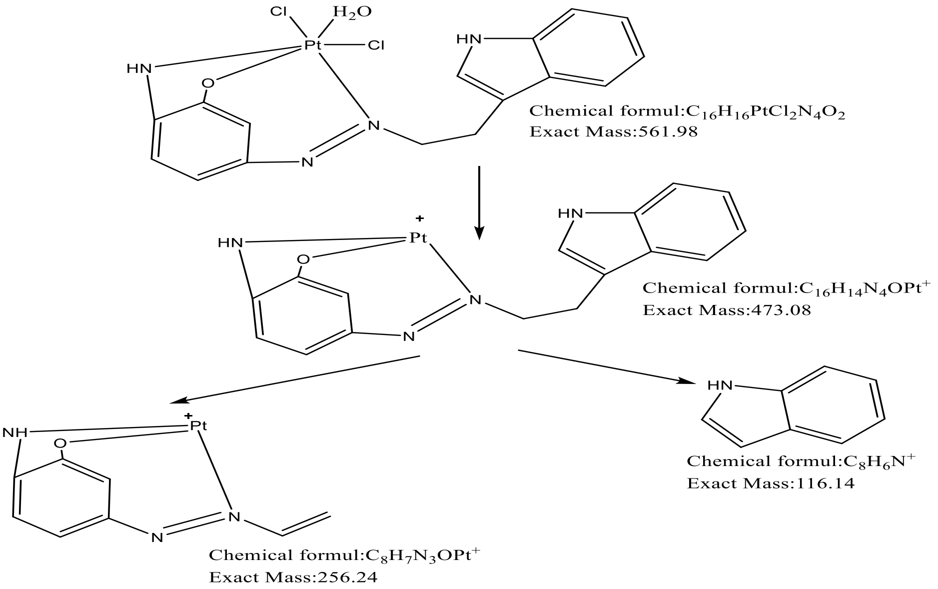
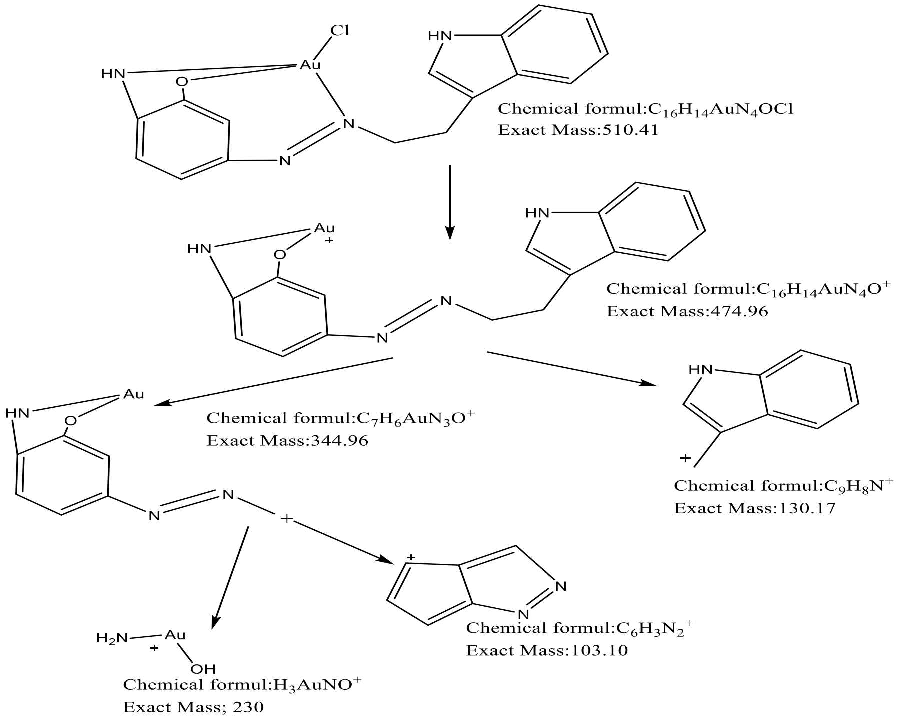


Figure 4.mass spectrum for Pt(IV) complex




H_2O
HN
Pd
O
N
N
HN
Chemical formul:$C_{16}H_{16}AuN_4O_2$
Exact Mass:402.42
Pd
+
HN
O
N
N
HN
Chemical formul:$C_{16}H_{14}PdN_4O^+$
Exact Mass:384.73
HN
Pd
O
N
N
+
Chemical formul:$C_7H_6PdN_3O^+$
Exact Mass:254.56
HN
+
Chemical formul:$C_9H_8N^+$
Exact Mass:130.17
+
N
N
Chemical formul:$C_6H_3N_2^+$
Exact Mass:103.10
H_2N
Pd
+
OH
Chemical formul:H_3PdNO^+
Exact Mass; 139.45


Cl
H_2O
Pt
Cl
HN
O
N
N
HN
Chemical formul:$C_{16}H_{16}PtCl_2N_4O_2$
Exact Mass:561.98
+
Pt
HN
O
N
N
HN
Chemical formul:$C_{16}H_{14}N_4OPt^+$
Exact Mass:473.08
+
Pt
NH
O
N
N
Chemical formul:$C_8H_7N_3OPt^+$
Exact Mass:256.24
HN
Chemical formul:$C_8H_6N^+$
Exact Mass:116.14



Scheme 2: Fragmentation Pattern for Ligand (L)and their complexes

3.3.(U.V–Vis) spectral data

The UV-Vis spectrum for ligands (L) and their complexes was observed in DMSO within the range of 200–1100 nm. **Figure 5** mostly shows two peaks where the ligand absorbs light at (284,421) nm. These peaks are related to π→π* and intra-ligand charge transfer (ILCT), respectively [16]. The electronic spectral of the Ni(II) complex shown in **Figure 6** spectra shows peaks at 276 nm and 410 nm, which relate to the π⟶π*, intra-ligand charge transfer (ILCT), and it shows two peaks at (579,989)nm with electronic transitions of type $^3T_1 \rightarrow {}^3T_{1F}$ and 3T1→3T1p,respectively. It supports tetrahedral geometry [17]. The UV-Vis spectra of the Pd (II) complex are displayed in **Figure 7**. The peaks at (286)nm nm are attributed to the π⟶π*, and (572)nm is related to the $({}^1A_1g \rightarrow {}^1B_1g)$, which demonstrates the geometry of a square planer. The electronic absorption of Au (III) complex assigned in peaks at (362) nm belongs to the π⟶π*, and (489) nm and (568) nm are ascribed to the${}^1A_1g \rightarrow {}^1B_1g$, ${}^{1A1g \rightarrow 1A2g,}$ respectively, which provide support for the square planer geometry [18–26]. **Table 3** provides the magnetic moments, electronic transition, and suggested formula of ligands (L) and their metal complexes.

Table 3. The UV-Vis spectra, magnetic moments for ligand (L) and its complexes

Comp.	**Wave number**		**ABS**	**ε max molar^{1}.cm^{1-}**	**Assignment**	**μ eff BM**	**Suggested Structure**
	(nm)	(cm^{-1})					
	284	35211.8	1.6	1600	$\pi\rightarrow\pi^*$	-	-
	421	23752.9	0.3	300	$n\rightarrow\pi^*$C.T L→L		
$[Ni(L)(H_2O)]$	276	36231.8	0.57	570	$\pi\rightarrow\pi^*$	3,01	tetrahedral
	410	24390.2	0.38	380	M→LC.T		
	579	17271.1	0.31	310	$^3T_1\rightarrow{}^3T_{1F}$		
	989	10111.2	0.01	10	$^3T_1\rightarrow{}^3T_{1p}$		
$[Pd(L)(H_2O)]$	286	34965.0	3.96	3960	$\pi\rightarrow\pi^*$	diamagnetic	square planer
	572	17482.5	0.27	270	$^1A_1g\rightarrow{}^1B_1g$		
$[Pt(L)(H_2O)Cl_2]$	246	40650.4	4.0	4000	$\pi\rightarrow\pi^*$	diamagnetic	octahedral
	375	26666.6	3.44	3440	M→LC.T		
	385	25974.0	3.94	3940	$^1A_1g\rightarrow{}^1T_1g$		
$[Au(L)Cl]$	362	27624.3	4.0	4000	$\pi\rightarrow\pi^*$	diamagnetic	square planer
	489	20449.8	1.18	1180	$^1A_1g\rightarrow{}^1B_1g$		
	568	17605.6	1.70	1700	$^1A_1g\rightarrow{}^1A_2g$		

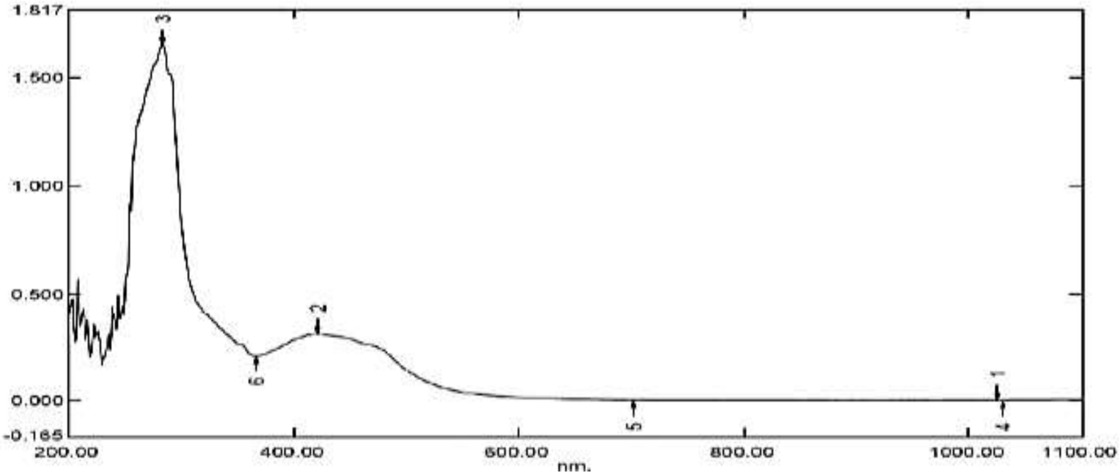


Figure 5. UV-Vis Spectra for ligand (L)

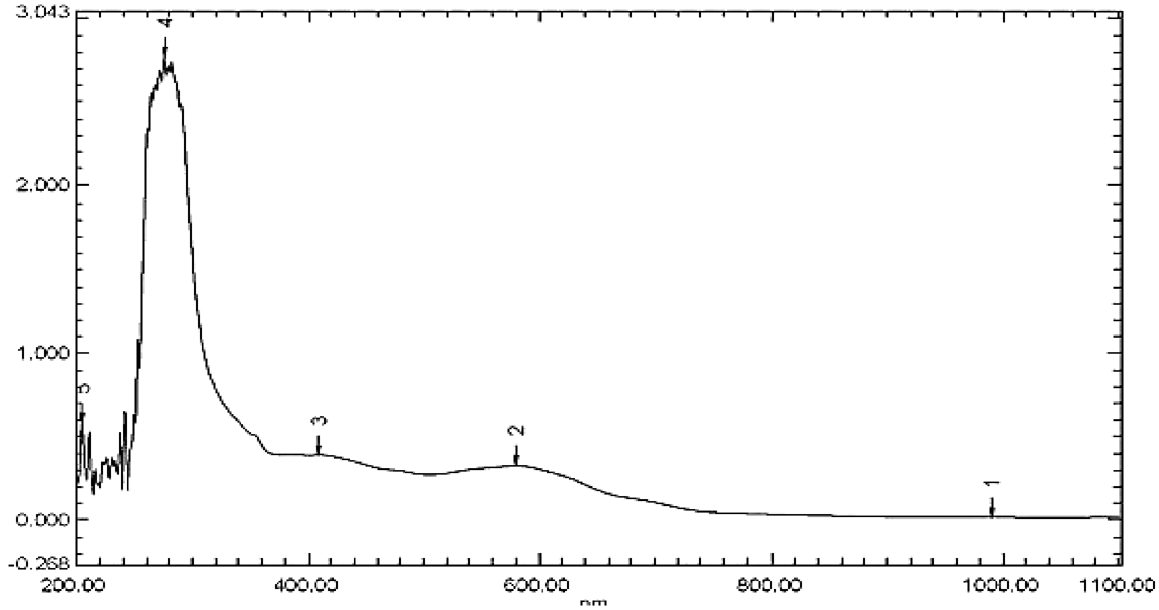


Figure 6.UV-Vis Spectra for[Ni(L) (H_2O)]

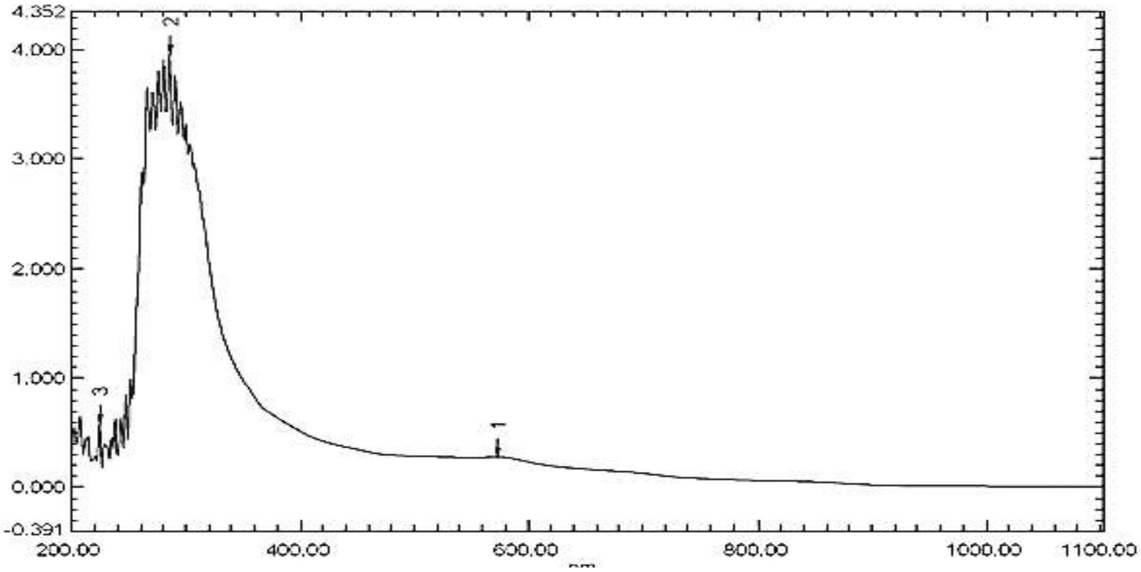


Figure 7.UV-Vis Spectrum for[Pd (L) (H_2O)]

3.4 FT-IR Spectral data

FTIR information was used to determine the functional groups of molecules that contain the donor atom when coordination takes place (especially in organics) [19, 20]. In comparison to the free raw materials, the ligand's (L) FTIR spectrum shows bands at (3408) cm-1 that were assigned to the stretching vibration for (NH2), (3759, 3286) cm-1 that belong to (O-H), and (NH) indole rings, respectively [21–27], and at 1485 cm-1 is attributed to the new azo group (N=N), which supports the ligand's formation. However, when compared to the presence of this vibrational mode in the free ligand, the FT-IR spectra for the complexes of Ni(II), Pd(II), Pt(IV), and Au(III) revealed that the stretching vibrational behavior of the O-H phenolic group had disappeared, indicating the presence of coordination through phenolic oxygen [23].

They also revealed that the N=N mode had changed in shape, intensity, and position when compared to the mode of ligand, the coupled water molecule with the metal ion was shown to exhibit stretching vibrational activity at 3512, 1611, and 743cm^{-1}, and at 3531, 1588cm^{-1}, and 744cm^{-1}, assigned to the υ(H_2O) aqua of the Ni(II) and Pd(II) complexes, respectively. This shows that the ligand and metal ion were coordinated via the ligand of the H_2O molecule [28–31]. The following **Table 3** contains a list of all Fourier transfer returns.

Table 4. The IR spectra bands (cm^{-1}) for ligand (L), and metal complexes

Comp.	ν (NH)	ν (C-H) arom.	ν (C-H) alipha.	ν (N=N)	ν (H_2O) aqua	Additional bands
L	-	3016	2918	1485	-	ν (NH_2) 3408 ν $(NH)_{indole}$3286 ν $(OH)_{phenolic}$3759
[Ni(L_2) (H_2O)]	4320	3198	2922	1458	3512 1611 743	-
[Pd (L_2)(H_2O)]	3409	3111	2966	1457	3531 1588 744	-

4. Thermal decomposition studies

The TGA thermal analysis was carried out to calculate the real loss of organic moiety present in the compounds. The ligand (L) and its metal complexes break down at high temperatures, as seen in **Figures 8–9**. **Tables 5, 6,** and **Scheme 2** give more details. The thermal decomposition process, Azo ligand (L), and its metal complexes were investigated. Utilizing thermogravimetric analysis at a heating rate of 10 ^{o}C/min in a nitrogen environment and temperatures ranging from 35 to 700 ^{o}C. The thermograms were used to calculate the weight loss percentages for compounds shown on the thermograms' decomposition stages, temperature ranges, and decomposition products. They found that there was an agreement between the estimated values from the thermal decomposition that was consistent; this confirms the results of the elemental investigation and validates the proposed suggested structure [32–34] from the DSC curves, and the thermodynamic parameters ΔH, ΔS and Δ G.

Table 5: Ligand (L)and their complexes TGA data

Compound	Ti C^o	T_f C^o	T max	% calculated (Estimated) Mass loss	Total Mass	Assignment
L	54.326	596.678	368.78	95.6050 (96.556)	95.6050 (96.556)	$-C_{15}H_{16}N_4O$
[Pt(L_2) (H_2O) Cl_2]	48.31 120.7 316.711	120.7 316.17 596.07	80.32 218.2 430	15.8270 (16.1680) 15.5389 (16.9720) 30.00545 (28.777)	61.4204 (61.917)	$-H_2O$ $-Cl_2$ $-C_6H_7N$ $-C_{10}H_7N_3$

Table 6. Ligand (L)and their complexes DCS data

Compound	T_i °C	T_f °C	Tmax ˚C	ΔH J/g	ΔS J	ΔG J	Type
L	33.78	92.45	78.17	-12.90	-0.21	3.5157	endothermic
	105.93	127.97	114.91	-1.39	-0.062	5.734	endothermic
[Pt(L) (H_2O) Cl_2]	43.14	60.31	56.51	-3.49	-0.203	7.981	endothermic
	60.86	98.24	76/73	-34.35	-0.918	36.088	endothermic
	172.41	257.21	224.98	93.84	1.106	-154.987	exothermic
	326.45	332.99	329.57	-0.75	-0.114	36.82	endothermic

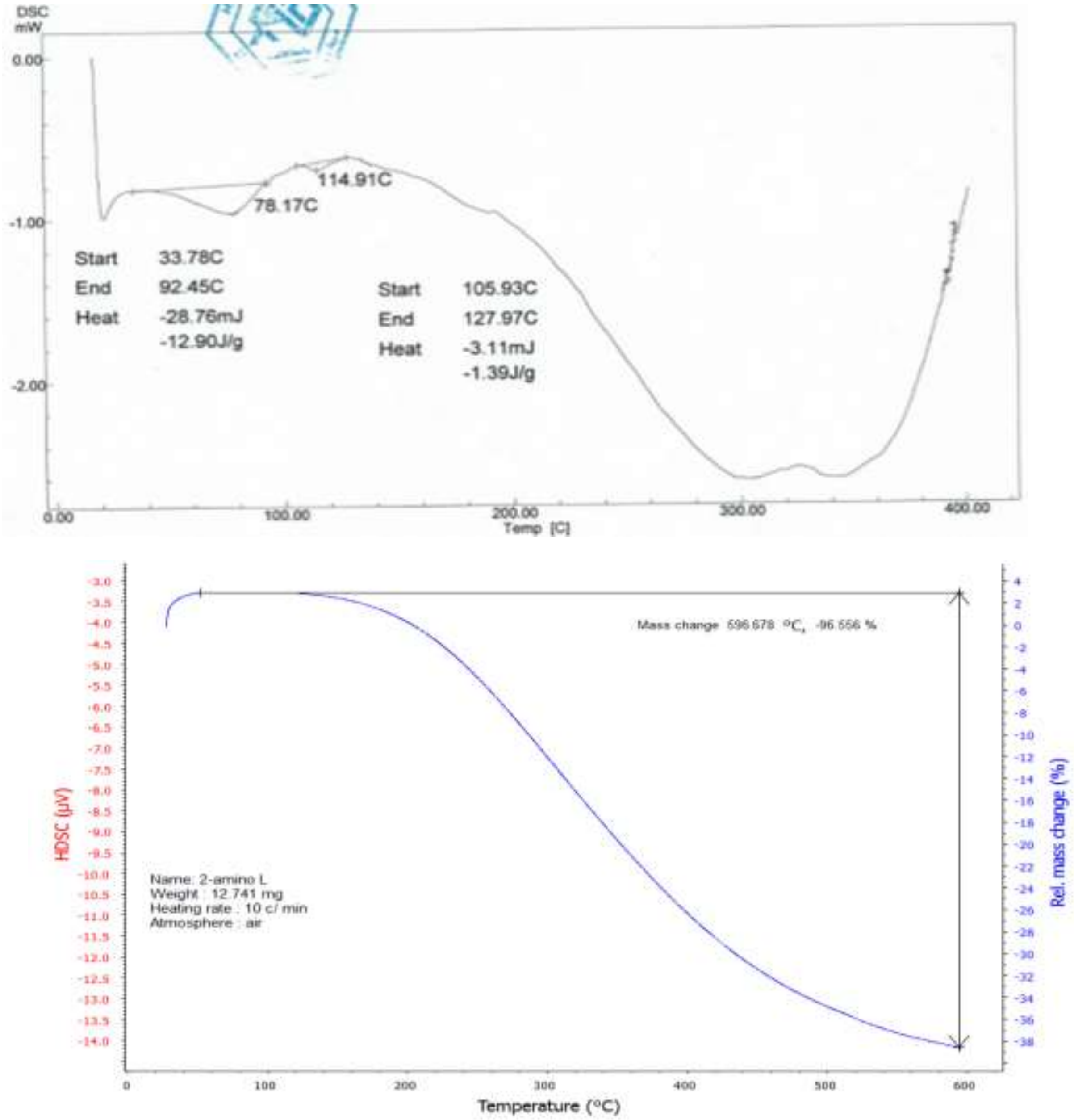


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Figure 8. Thermo gravimetric from Ligand

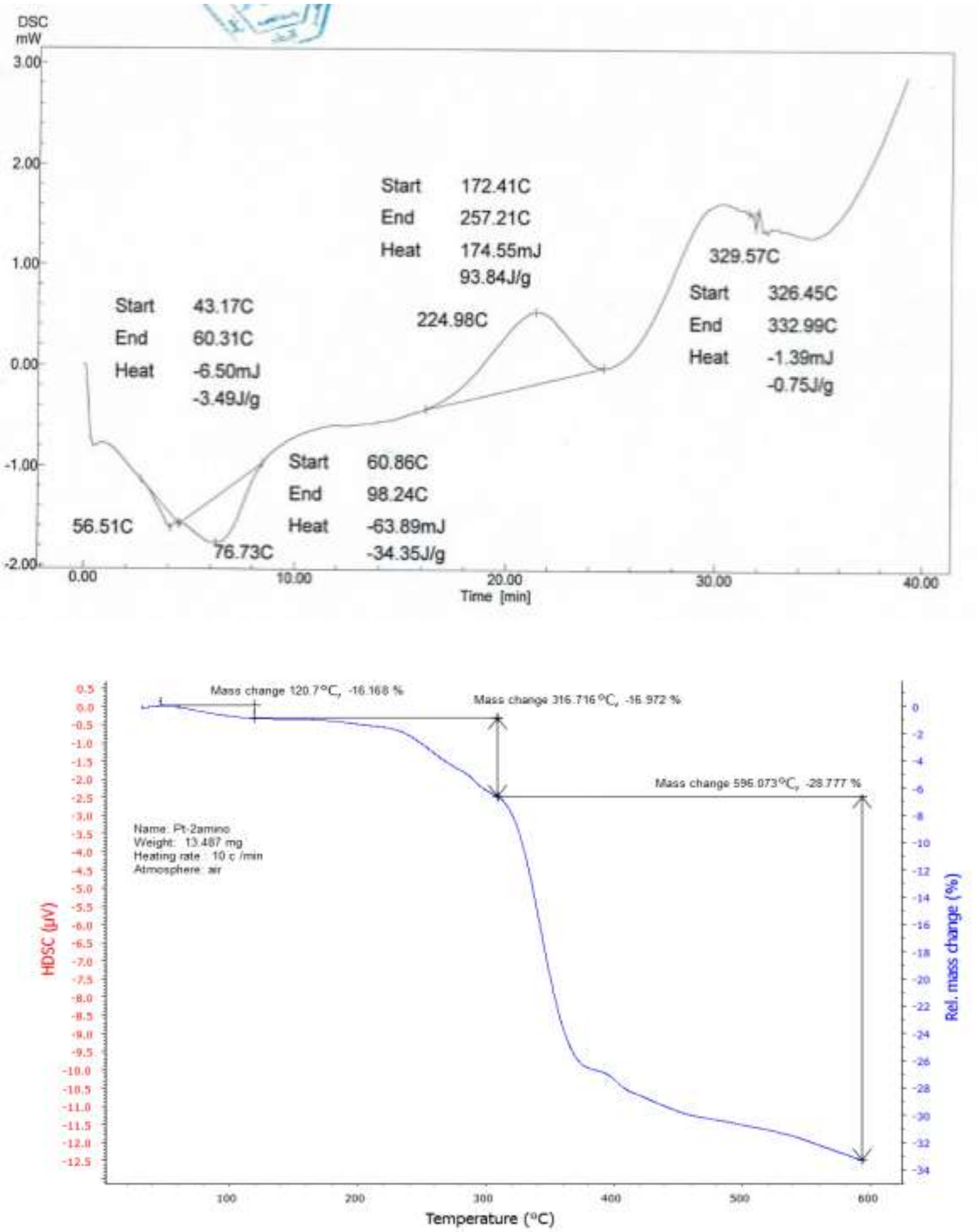
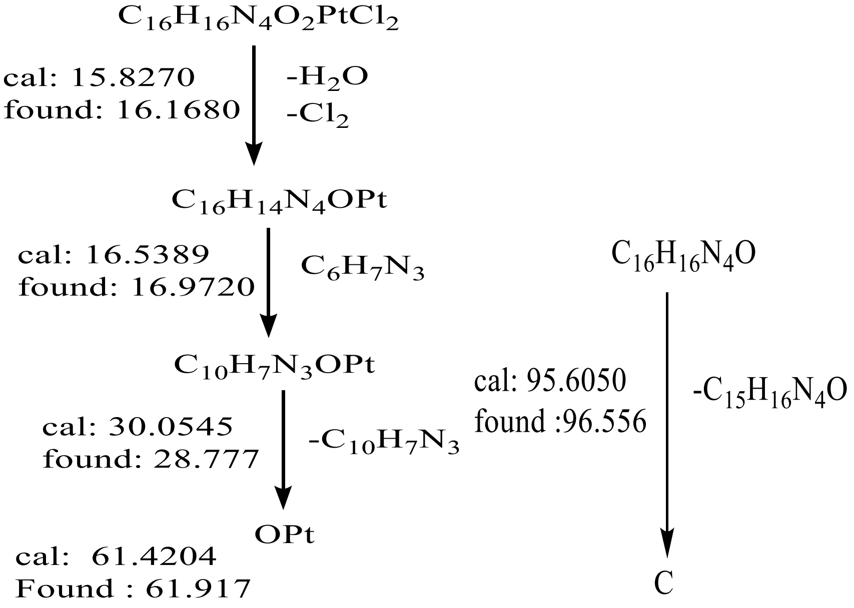


Figure 9. Thermo gravimetric from Pt(IV) complex



Scheme 2: Fragmentation pattern from ligand and Pt(IV) Complex

5. Antioxidant study

The results of each test of compounds were averaged. According to table 6, the interaction of compounds [Ni(L)(H_2O), [Pd(L)(H_2O)], and [Pt(L)(H_2O) Cl_2] with radicals resulted in a reduction of DPPH radical intensity. The radicals were subsequently scavenged by hydrogen donation [35–37]. The antioxidant activity of the ligand was higher than that of their metal complexes at 30 minutes. Gallic acid shows more antioxidant activity than ligand and its metal complex at 30 minutes; the Gallic acid and the ligand exhibit better scavenging activity at 30 minutes, while the Au(III) complex exhibits the least scavenging activity to compare Gallic acid [38-41].

Table 6: DPPH scavenging activities of prepared compounds at 30 Minute

Tested sample	Mean	Standard deviation	Coefficient of Variation%	Correlation Coefficient	IC_{50}
L	59.9671	2.0665	2.2221	0.9967	-6.0
[Ni(L)(H_2O)]	28.9867	11.9065	23.3256	0.9966	0.5443
[Pd(L)(H_2O)]	50.1516	2.2012	3.7766	0.3996	0.3531
[Pt(L)(H_2O) Cl_2]	54.3541	4.3322	13.6217	0.7671	0.9866
[Au(L) Cl]	60.6001	4.4765	14.4678	0.8866	1.5951
Gallic acid	98.5600	2.0846	2.2281	0.9966	-6.0304

IC_{50}: the half maximal inhibitory concentration.

6. Conclusion

In this study, we explain the synthesis of a new azo ligand and the full characterization of the ligand and its complexes. The Ni(II) complex was tetrahedral, while octahedral geometry suggests the Pt(IV) complex. Both Pd(II) and Au(III) complexes are square planar, which supports UV-Vis mass spectroscopy. The ligand (L) is a tridentate ligand and is coordinated by the metal ions through N.N.O. atoms, according to the results of the spectroscopic analysis of all complexes. The synthesized compounds' antioxidant properties were tested against the DPPH radical (1.1-diphenyl-2-picrylhydrazyl), and their results were compared to those of a common natural antioxidant called gallic acid. The outcomes demonstrate how successful the ligand was at scavenging free radicals. Synthesis, Characterization, Thermal Analysis, and Bioactivity of Some Transition Metal Complexes with New Azo Ligand.

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