Journal of Medicinal and Chemical Sciences 6 (2023) 121-131

Journal of Medicinal and Chemical Sciences

Journal homepage: http://www.jmchemsci.com/

Original Article

Synthesis, Characterization and Thermal Study of Some New Metal Ions Complexes with a New Azo 2-((2-(1*H*-Indol-2-yl)ethyl)diazinyl)-5-aminophenol

Myasim Qasim Abdulridha[1,*], Abbas Ali Salih Al-Hamdani[1], Waleed Ali Mahmoud[2]

[1]*Department of Chemistry, College of Science for Women, University of Baghdad, Baghdad, Iraq*
[2]*Dijlah University College, Prosthodontic Techniques Department, Baghdad, Iraq*

ARTICLE INFO

Article history

Receive: 2022-07-08
Received in revised: 2022-07-16
Accepted: 2022-07-26
Manuscript ID: JMCS-2207-1574
Checked for Plagiarism: **Yes**
Language Editor:
Dr. Fatimah Ramezani
Editor who approved publication:
Dr. Behrooz Maleki

DOI:10.26655/JMCHEMSCI.2023.1.15

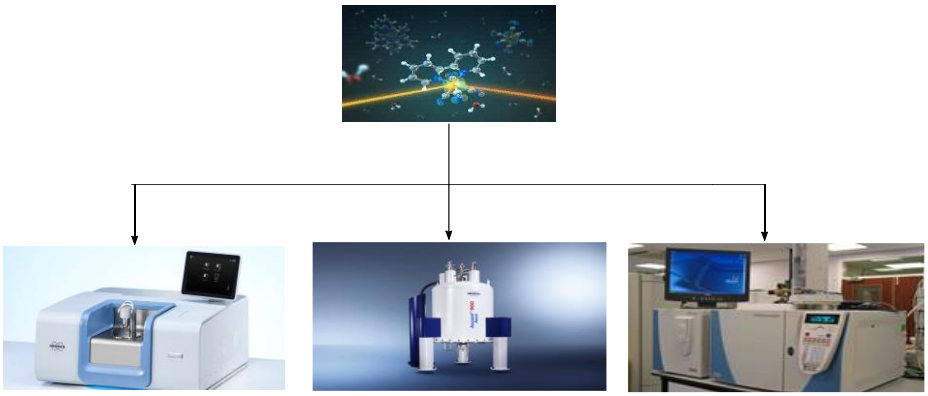
KEYWORDS

Azo ligand
Mass spectroscopy
Spectroscopic studies
Thermal study
2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol

ABSTRACT

This research includes the preparation of a new azo 2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol, in which the diazonium salt of 2(1*H*–Indole–3yl)- ethylamine reacts with a compound 3-aminophenol, and complexes have been prepared with the ions of Ni (II), Pd(II), Pt(IV), and Au(III). The characteristics of compounds include F.A.A, (C. H .N and O), ^{1}H & ^{13}C-NMR, IR, LC-Mass , UV-Vis spectral, DSC/TGA curve, the measurements of magnetic, and molar conductance. Each complex has an amount of [1:1] [M:L] and does not contain electrolytes. Based on the obtained results, molecular structural and geometry have been octahedral geometry suggest of Pt(IV) complex and tetrahedral of Ni (II)complex, the square planar of both Pd(II) and Au(III) complexes.

GRAPHICAL ABSTRACT



* Corresponding author: Myasim Qasim Abdulridha
✉ E-mail: Email: maysam.qasem1105a@csw.uobaghdad.edu.iq

Introduction

The N=N group is known as an azo group. Azo compounds are very significant groups of chemical molecules that attract the study attention. They are brightly colored and have long been used as dyes and paints. Furthermore, they have been extensively researched for uses like optical recording, oil-soluble lightfast dyes, and inkjet printing due to their superior thermal and optical properties [1]. Azo-dyes are commonly used dyes, accounting for around 60% of all dyes [2,3]. It is capable of bind with many compounds that have high density of electrons and consequently, it has an importance in estimation those compounds. Diazonium salts have an important role in dyes chemistry, so they bind with phenols, naphthol, and naphthyl amine forming many types of polychromic dyes [4]. They are prepared in two methods; either in a direct method by reducing nitro benzene compounds in presence of zinc or sodium acetate, this method is used for preparing symmetric azo benzene compounds [5]. The second type is known as indirect method by reducing nitro compounds to nitrous compounds by Diazotization reaction which can be defined as the reaction between sodium nitrite and conchydrochloric or sulfuric acid in a cold solution forming nitrous acid which in turn reacts with the aromatic amine to form diazonium salt that has electrophilic features which make it to be capable of binding with compounds that contain a high density of electrons such as amines or phenols leading to form azo compounds that are of high importance in manufacturing of dyes and pharmaceuticals in addition to their analytical importance in estimating many elements and compounds[6]. Diazonium salts forming from the reaction of primary aromatic amine with nitrite ion in acidic middle at 0-5 °C, this reaction called Diazotization reaction. Azo compounds will be prepared by the reaction of diazonium salt with coupling compound which always phenol ring or aromatic amine [7]. There are many important applications of Azo compounds, there were used in textile and carpet dyeing [8]. In Spectroscopic determination for minor concentrations of metal ions in ppm or less [9,10]. Azo compounds are used in extraction by solvent because of their low solubility in water compared with organic solvents, at which they soluble [11]. In this work, synthesis of a new azo 2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenoland was carried out. All complexes were formed and characterized by F.A.A, ^{1}H&^{13}C-NMR, LC-Mass, IR, and UV-Vis spectra. In addition, elemental micro analysis, chloride content, molar conductivity, magnetic susceptibility, and DSC/TGA curve were studied.

Materials and Methods

All chemical ingredients of 2(1*H*–Indole–3yl)-ethylamine ,3-aminophenol, conc. Were obtained. HCl, absolute ethanol, DMSO, $NaNO_2$, NaOH, salts of metal ($NiCl_2.6H_2O$), ($H_2PtCl_6.6H_2O$), ($PdCl_2$), and ($HAuCl_4$) were obtained from Sigma-Aldrich, Merck, and others.

Euro vector model EA/3000, single-V.3.O-single was used to conduct elemental analyses (C, H, N, S, and O). Metal ions were estimated as metal oxides by using a gravimetric method. The molar conductance of the complexes was measured by using a Conductometer WTW at 25 °C at a concentration of 1×10^{-3}M. Also, DMSO was used to dissolve all of the complexes (DMSO) on a mass spectrometry (MS) QP50A: DI Analysis Shimadzu QP-2010-Plus (E170Ev) spectrometer. In addition, the spectra in the (UV-Vis) region were investigated by using the UV-V spectrophotometer UV-1800 Shimadzu. A Brucker 400 MHz was used to record (^{1}H & ^{13}C-NMR) spectra of ligand in DMSO-d_6. The IR Prestige-21 was used to investigate the Fourier transform infrared (FTIR) spectra, and the Perkin-Elmer Pyris Diamond TGA&DSC was used to conduct thermogravimetric studies.

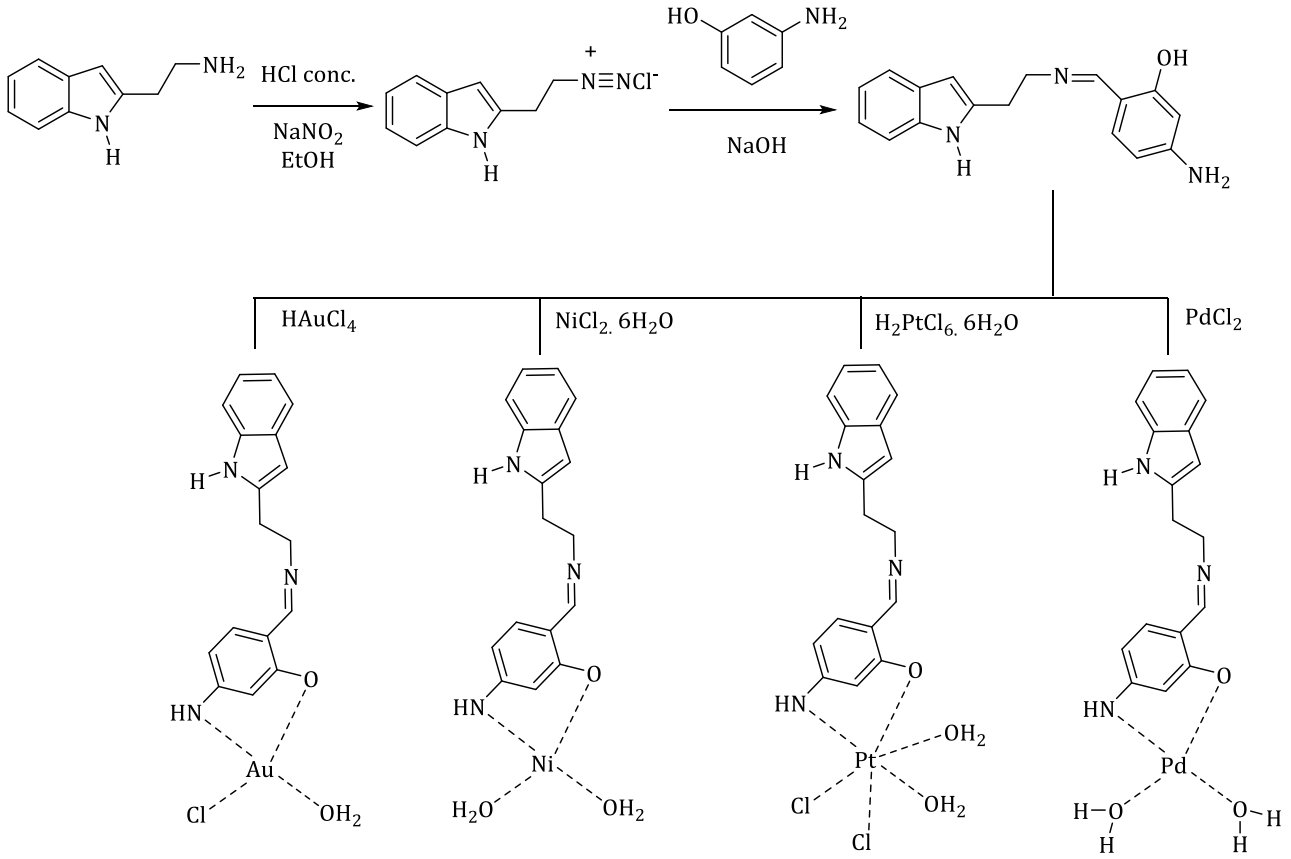
Synthesis of 2-((2-(1H-indol-2-yl)ethyl)diazinyl)-5-aminophenol

2(1*H*-Indole - 3yl)- ethylamine (1 g, 0.006 mol) was dissolved in 2 mL HCl, 10 ml of ethanol at 5 °C, while the mixture was refrigerated, and then to avoid temperature increases of up to 5 °C, (10%, 0.43 g, 0.006 mol) hydrated sodium nitrite $NaNO_2$ was gradually added. Next, 0.671 g, (0.006 mol) of 3-aminophenol dissolved was added to 10 ml of ethanol after the reaction has been stirred for around 30 minutes. The result developed a dark

brown hue, was filtered, allowed to dry, collected, and then weighed. It had a yield of 73 % and a melting point of 171–173 °C [12]. The processes of the diazotization coupling are depicted in Scheme 1.

Synthesis of Complexes

A metal salt solution $NiCl_2.6H_2O$ (0.23 g, 0.001 mol), $PdCl_2$ (0.19 g, 0.001 mol), $H_2PtOCl_6.6H_2O$ (0.37 g, 0.001 mol), and $HAuCl_4$(0.37 g, 0.001 mol) in 10 mL of ethanol, and a solution of the ligand (0.31 g, 0.001 mole) were added to 10 ml of ethanol with the amount of [1:1] M:L. Next, the mixture was refluxed for 2 hours at (50-70 °C). After that, it was cooled in an ice bath until precipitation started, and overnighted away from any complexes. The precipitates were filtered and a little quantities of heated ethanol was used to eliminate any traces of unreacted components, and then it was dried by using vacuum desiccators over mixed $CaCl_2$, which was weighed after being gathered. Table 1 lists the physical characteristics of ligand and its complexes.



Scheme1: Synthesis of ligand and their complexes

Results and Discussion

The physical characteristics and elemental results obtained from (C.H.N.O.) analyses, chloride contents, and metal contents of the prepared compounds are described in Table 1. The experimental results showed an agreement with the theoretical value. It confirmed the suggested formula.

^{1}H-NMR spectra

The 2-(2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol^{1}H-NMR spectrawere measured in DMSO-d_6 as the solvent and using TMS as an internal reference, as shown in Figure S1. The ligand has undergone studies and is listed in Table 2 [12-14].

Table 1: Analytical and physical data of ligand and its metal complexes

Compound	M.f M.wt	%C (Expert) theoretical	%H (Ekjhgxpert) theoretical	%N (Expert) theoretical	%O (Expert) theoretical	%M (Expert) theoretical	%Cl (Expert) theoretical	Color	m.p. °C	%Yield	Molar conductivity. $Ohm^1\ cm^2\ mol^1$
L	$C_{16}H_{16}N_4O$ 280.32	(69.12) 67.84	(5.55) 6.71	(20.98) 19.78	(6.17) 5.65	-	-	Dark Brown	171-173	73	-
$[Ni(L)(H_2O)_2]$	$C_{16}H_{18}N_4NiO_3$ 373.07	(50.97) 51.52	(4.55) 4.83	(15.88) 15.03	12.52 12.87	(16.11) 15.78	Nil	Dark Green	d217-220	72	18
$[Pd(L_1)(H_2O)_2]$	$C_{16}H_{18}N_4PdO_3$ 420.76	(44.79) 45.66	(4.58) 4.28	(14.33) 13.32	(11.67) 11.41	(24.67) 25.31	Nil	Redish Brown	d287-285	60	22
$[Pt(L)(H_2O)_2Cl_3]$	$C_{16}H_{18}N_4PtO_3Cl_2$ 580.32	(32.17) 33.09	(3.82) 3.10	(10.56) 9.65	(7.85) 8.27	(34.05) 33.62	(11.63) 12.23	Pink	d291	62	20
$[Au(L_1)(H_2O)Cl]$	$C_{16}H_{16}AuClN_4O_2$ 528.74	(35.68) 36.33	(3.11) 3.02	(11.56) 10.59	(5.98) 6.05	(36.56) 37.27	(7.21) 6.70	Light Gray	d301	71	18

d= decompose

Table 2: ^{1}H-NMR spectral data for 2-(2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol

Chemical shift δ (ppm)	Functional Group
2.50	DMSO
6.78	((1H) s,CH-NH Indole)
6.78-7.80	((1H) s,CH-N=N)
6.75	((1H) d,CH-OH)
7.80	((4H) m, CHarom)
9.51	((2H) s,NH_2)
10.5	((1H) s,OH)
11.30	((1H) s,NH)

^{13}C-NMR Spectra

Several chemical shifts have been identified by the ^{13}CNMR Spectra (160.16, 173.78, 131.89, 137.21, 157.34, -181.32, 48.10, 34.70, 129.80, 165.11, 149.83, 145.12, 154.12, 117.44, 179.20, and 170.1) to the carbon atoms at the sites (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, and 16), respectively. Figure S2 depicts the ^{13}CNMR of the

2-(2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol.

Electronic transition

The electronic absorption spectra of 2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol are demonstrated in Figure 3 which indicates absorption peaks at (278 nm, 35971.2 cm^{-1}, max 15100 $L.mol^{-1}.cm^{-1}$) attributable to the π⟶π* and (361 nm, 27700.8 cm-1, max 580 $L.mol^{-1}.cm^{-1}$) due to the n⟶ π* [15,16].Ni(II) complex. The electronic absorption spectra showed peaks at 284 nm and 443 nm ascribed to the π⟶π*, (C. T) and revealed two peaks at (618 and 677) nm electronic transitions type $^3T_1 \rightarrow {}^3T_{1F}$, $^3T_1 \rightarrow {}^3T_{1p}$, respectively. It was a good evidence for tetrahedral geometry [17]. The Pd (II) complex is illustrated in Figure 4. The peak at (285) nm is ascribed to the π⟶π* and 310 nm is attributed to the (C.T) which displayed two new absorption peaks at (458) nm and (562) nm of electronic transitions type $^1A_1g \rightarrow {}^1B_1g$ and $^1A_1g \rightarrow {}^1A_2g$, respectively, this is indicative of square planer geometry [18,19]. The peaks at (283 and 322) nm are ascribed to π⟶π* and (C.T). In addition, two new absorption peaks at (456 and 575) nm are attributed to transitions type $^1A_1g \rightarrow {}^1T_2g$, $^1A_1g \rightarrow {}^1T_1g$, respectively of Pt(IV) complex. It was indicative of octahedral geometry [20,21]. The electronic absorption of [Au (L)(H_2O)Cl] peaks at (285) nm is ascribed to the π⟶π*, and (442) nm, (578) nm are ascribed to the $^1A_1g \rightarrow {}^1B_1g$, $^1A_1g \rightarrow {}^1A_2g$, respectively, which are a good evidence for square planer geometry [22], electronic transition, magnetic moments, and suggested formula of metal complexes given in Table 3.

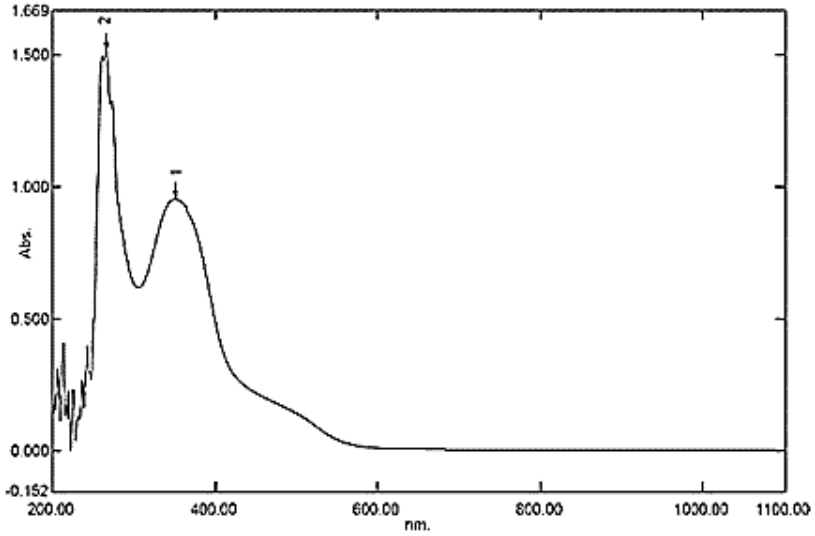


Figure 1: Electronic spectra of ligand (L)

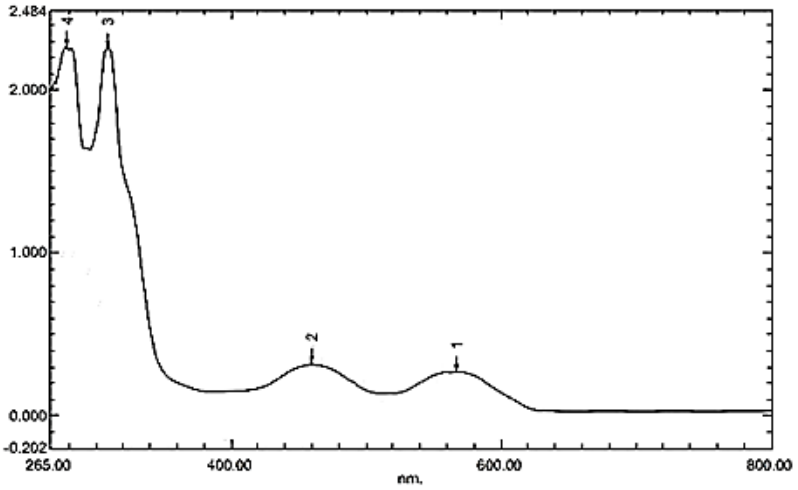


Figure 2: Electronic spectra of [Pd (L)(H_2O)$_2$]

Table 3: Electronic transition, magnetic moments, and suggested formula of metal complexes

Compounds	Wave (nm)	Number (cm^{-1})	ABS	ε_{max} L $mol^{-1}cm^{-1}$	Transition	μ_{eff} (BM)	Suggested formula
L	278 361	35971.2 27700.8	1.51 0.58	15100 580	π⟶π* n→π*, L→LC.T	-	-
[Ni(L) $(H_2O)_2$]	284 370 443 618 677	35211.2 27027 22573.3 16181.2 14771	2.28 1.92 0.17 0.12 0.14	2280 1920 170 120 140	π⟶π* n→π*.L→LC.T $^3T_1 \rightarrow {}^3T_{1F}$ $^3T_1 \rightarrow {}^3T_{1p}$ $^3T_1 \rightarrow {}^3A_2$	3.88	Tetrahedral
[Pd (L)$(H_2O)_2$]	287 310 458 562	34843.2 32258 21834 17793.5	2.28 2.30 0.35 0.38	2280 2300 2350 380	π⟶π* M→L C. T $^1A_1g \rightarrow {}^1B_1g$ $^1A_1g \rightarrow {}^1A_2g$	Diamagnetic	Square planer
[Pt(L) $Cl_3(H_2O)$]	283 322 456 575	35335.6 31055.9 21929.8 17391.3	2.28 2.15 0.28 0.30	2280 2159 280 300	π⟶π* M→L C. T $^1A_1g \rightarrow {}^1T_2g$ $^1A_1g \rightarrow {}^1T_1g$	Diamagnetic	Octahedral
[Au (L)(H_2O)Cl]	285 442 578	35087.7 22624.4 17301	2.28 0.29 0.31	2280 290 310	π⟶π* $^1A_1g \rightarrow {}^1B_1g$ $^1A_1g \rightarrow {}^1A_2g$	Diamagnetic	Square planer

LC-Mass Spectral

The mass spectrum of the ligand 2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol and Ni(II), Pt(IV), Au(III) complexes is given in Figures S3 and S4, mass fragment ion are displayed in Scheme S1 [23-25].

Infrared Spectral Studies

The functional groups of molecules were identified by using FTIR data (that have the donor atom) when coordination occurs (especially organics) [26]. The FTIR spectrum ligand is indicated in Figure 4 that shows bands at (3460, 3427, and 1697) cm^{-1} were ascribed to the stretching vibration asυ(NH_2), sυ(NH_2), and δ(NH_2), 3761 and 3158 cm^{-1} that assign to υ(O -H), (NH) indole ring, respectively [27], and at 1471 cm^{-1} is attributed to the new azo group (N=N) compared with the free raw materials, which confirms the formation of the ligand [28,29]. Additionally, complexes are identified, and their spectra are contrasted with the spectrum of a free ligand. When compared with the ligand spectrum, all complex spectra show the removal of the (O -H) phenolic and (NH_2) bands. This proves that the ligand was coordinated with the metal ion through the nitrogen and oxygen atoms [30] and new bands are appeared that belongs to (M-N) at (561, 533, and 549) cm^{-1} for the complexes (Ni, Pd, and Pt), respectively, (M-O) at (499, 480, and 493) cm^{-1} for the complexes (Ni, Pd, and Pt), respectively which supports coordination occurrence through the nitrogen and oxygen atoms [31,32]. All complexes showed new bands to correlate to coordinated water molecules in the complexes [33]. Characteristic vibrations and assignments of the ligand and its complexes are reported in Table 4.

Table 4: IR (in cm^{-1}) studies of the ligand (L) and its complexes

Compounds	ν(NH)	ν(C-H) aromatic	ν(C-H) aliphatic	ν (N=N)	ν (M-N)	ν (M-O)	ν(H_2O) aqua	ν (M-Cl)
L	-	3041	2927	1471	-	-	-	-
Ni(L) $(H_2O)_2$]	3394	3031	2914	1460	561	466	3564,1608 754	333
[Pd (L)$(H_2O)_2$]	3444	3002	2862	1456	553	480	3759, 1612 754	-
[Pt(L) $Cl_3(H_2O)$]	3296	3010	2852	1459	549	493	3734, 1623 740	329
[Au (L)(H_2O)Cl]	3395	3045	2978	1448	511	451	3558, 1600 770	335

Thermal Studies

By using (DSC/TGA) techniques, the results of the thermal and weight analyses of the compounds were determined to the compounds' stability in thermal and weight. It supports the formulae that have been proposed. We observed that the remaining complexes are metal oxides, and the remaining ligand is carbon. In addition, the DSC curve is used to determined exothermic, or endothermic and amount of heat [34- 36]. The results of thermal studies of the ligand and its complexes are represented in Table 5, Scheme 2 and Figures 3-5.

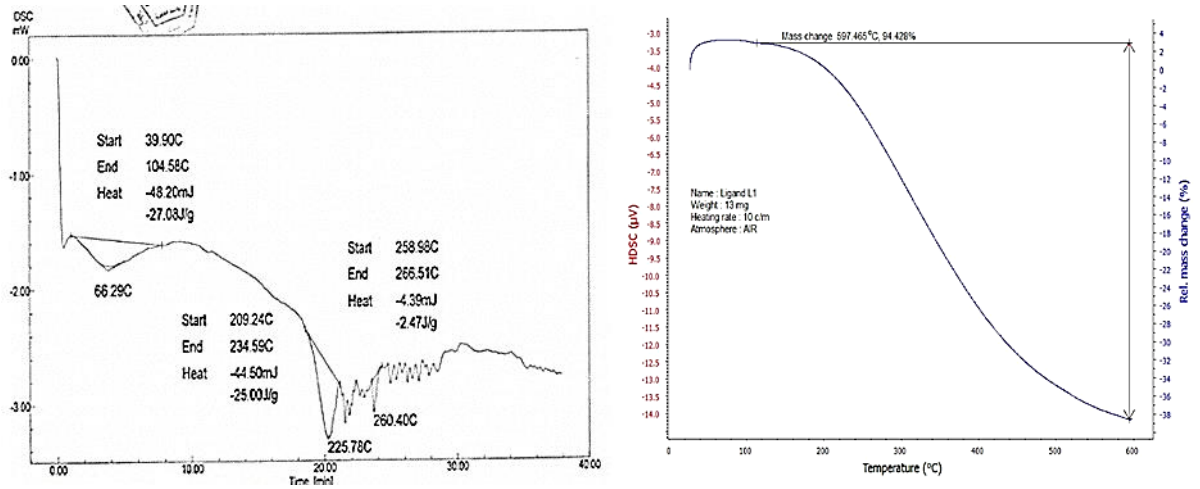


Figure 3: Thermal gravimetric for ligand

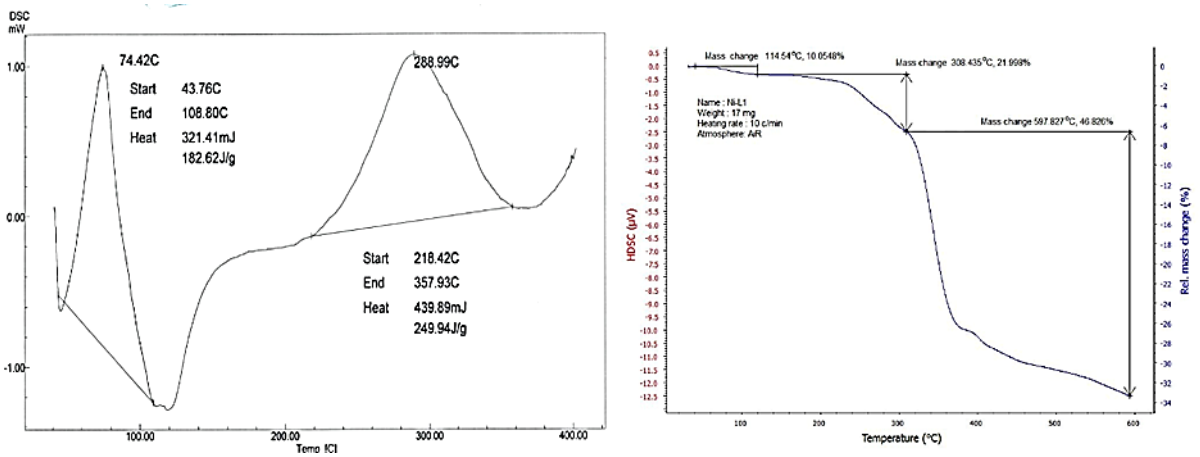


Figure 4: Thermal gravimetric for **[**Ni(L) $(H_2O)_2$]

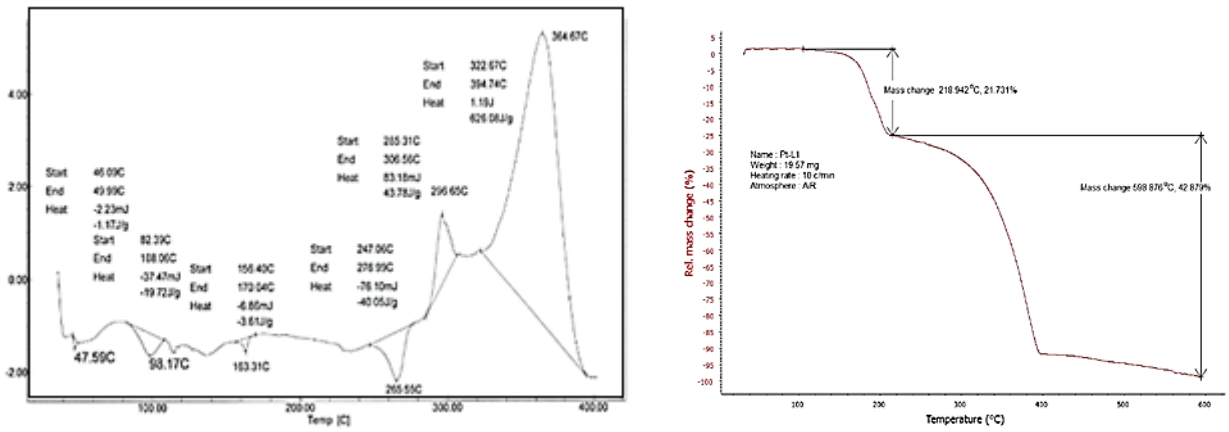
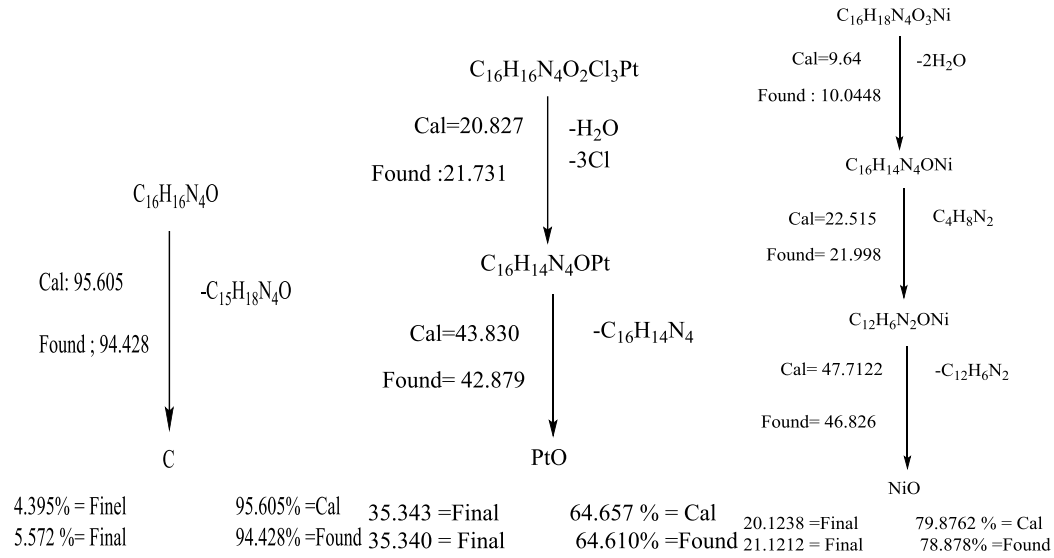


Figure 5: Thermal gravimetric for [Pt(L) $C_{l3}(H_2O)$]



Scheme 2: Potential decomposition reaction of complexes

Table 5: Thermal studies data for ligand and its metal complexes

Compounds	DSC max °C	Ti °C	T_f °C	Max TDTG	% Estimated (calculated)		Assignment
					Mass loss	Total mass loss	
L	66.29 endo 225.78endo 260.30endo	113.078	597.465	305.11	95.605 (94.428)	95.605 (94.428)	-C_5 $H_{18}N_4O$
[Ni(L) $(H_2O)_2$]	74.42exo 288.99exo	49.652 114.54 308.435	114.54 308.435 597.827	76.42 224.21 478.56	9.649 (10.0548) 22.51 (21.998) 47.7122 (46.826)	79.8762 (78.878)	-$2H_2O$ -$C_4H_8N_2$ -$C_{12}H_6N_2$
[Pt(L)$Cl_3(H_2O)$]	47.59endo 98.17endo 163.31endo 265.55endo 296.65 exo 364.67 exo	104.621 218.942	218.942 598.876	124.43 405.12	20.827 (21.731) 43.830 (42.879)	64.657 (64.610)	-H_2O -3Cl -$C_{16}H_{14}N_4$

Conclusion

This work investigated the synthesis and characterization of ligand 2-((2-(1*H*-indol-2-yl)ethyl)diazinyl)-5-aminophenol metal complexes. The complexes were produced by treating the new Azo ligand with various metal ions. The ligand was identified by using spectroscopic methods (UV-Vis, FTIR, ^{1}H and ^{13}C-NMR, and LC-Mass), the elemental microanalysis, and thermal studies (TGA/ DCS). The complexes were diagnosed by infrared, LC-Mass UV-VIS spectral methods, TGA, DSC curve, atomic absorption, elemental microanalysis, and molar conductivity. According to the molar conductivity results, it was found that all the prepared complexes are non-electrolyte, the IR spectrum of ligand was compared with the metal complexes to determine the coordination sites of N and O atoms. All the complexes had [1:1] M:L ratio. According to the results, molecular structure and geometry have been determined to be square planar for Pd(II) and Au(III) complexes, octahedral for Pt(IV) complex, and tetrahedral for Ni(II).

Funding

This research did not receive any specific grant from fundig agencies in the public, commercial, or not-for-profit sectors.

Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

There are no conflicts of interest in this study.

ORCID:

Abbas Ali Salih Al-Hamdani
https://www.orcid.org/0000-0002-2506-986X

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HOW TO CITE THIS ARTICLE

Myasim Qasim Abdulridha, Abbas Ali Salih Al-Hamdani, Waleed Ali Mahmoud. Synthesis, Characterization and Thermal Study of Some New Metal Ions Complexes with a New Azo 2-((2-(1*H*-Indol-2-yl)ethyl)diazinyl)-5-aminophenol. *J. Med. Chem. Sci.*, 2023, 6(1) 121-131

https://doi.org/10.26655/JMCHEMSCI.2023.1.15

URL: http://www.jmchemsci.com/article_154643.html