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Spectrophotometric, thermal and determination of trace amount of palladium (II) nickel (II) and silver (I) by using pyrazolone azo derivative

Abstract

This study describes the use of novel azodyes reagents, H_4L,1-[(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl]-2,3,7,8-tetrahydroxychromeno(5,4,3-*cde*) chromene-5,10-dione, that prepare ellagic acid as a coupling agent with 4-aminoantipyrine, as sensitive and selective analytical reagent for the determination of palladium (II),nickel (II) and silver (I) Ions spectrophotometrically. HL reacts with ions at pH (3,8 and 6) respectively to form colored complexes of 1:2 stoichiometry of Pd(II), Ni(II) and 1:1 for Ag(I) which show a maximum absorbance of 504, 495 and 492nm of molar ratio absorbitivity $3.565x10^3$, $1.966x10^3$ and $4.735x10^3L.mol^{-1}cm^{-1}$ respectively. Fixed optimum conditions were constructed, precision, accuracy, R.S.D.andcorrelation factors. The Beers law was obeyed 0.1-3ppm. The thermal measurements were also studied, all the complexes show three-stage mass loss in their TG/HDSC curves.

Keywords: spectrophotometric, azodyes, thermal analysis, palladium (II), nickel (II) and silver (I)

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Introduction

Azo compounds are very important class of chemical compounds containing a hetro cyclic moiety which has attracted the attention of many researchers in the recent years.[1] There has been increasing interest in syntheses of heterocyclic compounds that have biological and commercial importance clinical,[2] and pharmacological activities.[3] One of the most important derivatives of antipyrine is 4-aminoantipyrine, which is used as a synthetic intermediate to prepare polyfunctionally substituted heterocyclic moieties.[4]

Azo compounds containing N_3 donor atoms act as superior chelating agents for the transition and non-transition metal ions and showed biological activities.[5] Azo days are commonly synthesized by coupling a diazonium reagent with an aromatic compound to form an azo reagent.[6] The azo compounds give bright, high intensity colors, much more than the other most common compounds, in addition, they have fair to good fastness properties, their biggest advantage it their cost–effectiveness, which is due to the processes involved in manufacture.[7] The coordinating property of 4-aminoantipyrine ligand has been modified to give a flexible ligand system, formed by condensation with variety reagents like aldehydes, ketones and carbazides.[8]

Ellagic acid, a dimeric derivative of gallic acid, is apolyphenolic antioxidant that occurs in its free form as aglycoside or is found as ellagitannins in fruits and nuts ofseveral plants.[9] Several studies have reported the antioxidant, antimutagenic, anti-inflammatory and cardioprotective activityof ellagic acid.[10] In this research, novel sensitive and selective azodyes was prepared by reaction 4-aminoantipyrine with ellagic acid as a coupling agent to determine trace amount of Pd (II), Ni (II) and Ag (I) as complexes.

Experimental

Materials and methods

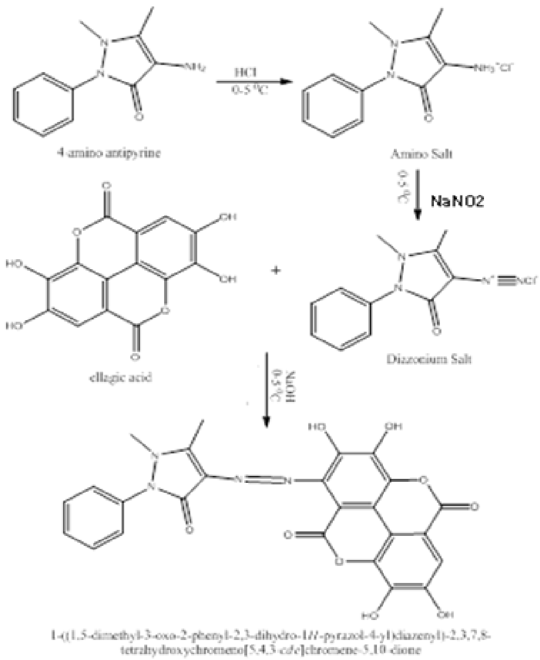
All chemicals ofhighest purity were used in this work which supplied by Fluka and BDH. Spectrophotometric measurements were made with ShimadzuUV-Visible-1650Pc double beam. The FTIR measurements were made in Shimadz 8400 Series Japan. Differential Scanning Calorimeter DSC MODEL STA PT-1000 Linseis. Atomic Absorption Spectrophotometer Flame Model Shimadzu AA-7000F. Electricmolar conductivity measurements were carried out at room temperature usingan Alpha digital conductivity model-UK 9300. The pH measurements were performed with a “HANNA pH meter H19811-5 Instrument.

Synthesis of H_4L

The reagents and solvents were of analytical grade and used without further purification. 4-Aminoantipyrine (0.00492mole) 1.0000gm was diazotized by dissolving it in 25ml ethanol then 5ml of HCl was adding keeping the temperature at 0-5°C and then adding $NaNO_2$ solution gradually and left the solution about 15min.[11] The diazonium salt was spontaneously added slowly drop wise to a well cooled alkaline solution of coupling agent (ellagic acid 1.4869gm), the mixture was allowed to stand for 1h. The dark colored mixture was neutralized with HCl and the solid precipitate was filtered off and washed several times with (1:1) (methanol: water) mixture then recrystallised from boiling methanol and left to dry (Scheme 1).

Solutions

A stock standard Palladium solution (100ppm) was prepared by dissolving 0.0353g of $PdCl_2$, the volume was completed to 200ml with distilled water. A stock standard Nickel solution (100ppm) was prepared by dissolving 0.0806 gm of $NiCl_2.6H_2O$, the volume was completed to 200ml with distilled water. A stock standard Silver solution (100ppm) was prepared by dissolving 0.0314gm of $AgNO_3$, the volume was completed to 200ml with distilled water. Solution of azodye reagent ($1x10^{-3}M$) were prepared by dissolving (0.0516gm) and complete the volume to100ml with absolute methanol. Foreign ions solutions (100µg ml^{-1}), these solutions were prepared by dissolving an amount of the compound in distilled water completing the volume in a volumetric flask.



Scheme 1 Synthesis of H_4L

Procedure

To get highest absorbance of complexes formed, it is necessary to get optimum conditions of forming each complex, which include, the selection of the suitable wavelength (λ_{max}), effect of time, effect of pH values, effect of sequence of additions, stoichiometry and effect of interferences of strange ions. The general procedure was summarized by taking (0.1–3ppm) of Pd(II), Ni(II) and Ag(I) ions with (3ml) of $1x10^{-3}$M of H_4L then the volume was completed to 10ml. After 15 min, the absorbance of formed complexes were measured at λ_{max} of 504, 495and492nm respectively.

Results and discussion

Absorption spectra

The electronic spectra of H_4L and their complexes with ions, Pd(II), Ni(II) and Ag(I). Complexes are at λ_{max} wavelengths were fixed in Figure 1, shows the absorbance λ_{max} at 504, 495 and 492nm respectively. It is clear that according to the red shift that happened in λ_{max} show the stable complexes are formed immediately, $\pi \longrightarrow \pi^*$ transition within the azo group and heterocyclic moieties involving the whole π electronic system of the compound influenced by inter-molcular charge transfere character.[12] A great bathochromic shift in the visible region was detected in the complex solution spectra with respect to that of the free ligand. The intensity colored solutions formed from the reaction of the azo ligands with the metal ions is playing an important rule for many UV-Vis spectral studies. This is because of the presence of a sharp and high absorption peak which belongs to the metal complex. The large bathochromic shift of this peak in the visible region with respect to that of the ligand may give a good indication on the complex formation.

FTIR spectra

The FT-IR data of H_4L reagent and their complexes are with their probable assignment given in Table 1. The important bands observed in the spectra of the complexes while comparing with reagents, which is helpful in detection of donation sites. The IR spectra of the free ligand have a broad band around 3415.9cm^{-1} which could be attributed to O-H stretching vibration and it shifted to lower frequency. The stretching frequency of carbonyl group of ligand ν(C=O) 1676.14(*s*) cm^{-1} is shifted to a lower frequency in complexes. Similarly the frequency corresponding to ν(N=N) at 1494.8 (*s*) is shifted to range (1383-1496)cm^{-1} in complexes. The shifting in λ_{max} and their intensities of bands led to predict the chelating behavior, i.e., coordination occurs through ring carbonyl oxygen atom with the nitrogen atom of azo group. The spectra of metal complexes also show additional bands in (546-563) *(w)* cm^{-1} and (480-418) *(w)* cm^{-1} which is probably due to the formation of ν(M-O) and ν(M-N)bond respectively.[13]

Table 1 The most important band and their assignments of H_4L and their complexes (cm^{-1})

Compound	νOH	νC-H Aromatic	νC=O	νN=N CNNC	νCN=NC	ν(C-N)	ν(M-O)	ν(M-N)
H_4L	3415.9s	3194.12m	1676.14s	1535.34m 1494.8s	1109.07m 1053.13m	------	------	------
Pd-L	3417s	3128.04m	1649.19s	1553.5w 1491.02m	1132.25s 1111.03s	1400m	534.3w	480w
Ni-L	3408.22s	3227.94w	1627.92s	------- 1496.76w	1151.5m 1118.71m	1406.11m	513.07w	414.70w
Ag-L	3423.76s	------	1651.12s	1514.17m 1383s	1136.11w 1030.02w	------	513.08w	450w

s, strong; m, medium; w, weak.

Effect of pH: The influence of pH value on the absorbance of complexes was studied at different pH (Figure 2) by using of HCl and NaOH (0.05M) solutions (pH 2-10). It was found that the highest absorbance of Pd-L, Ni-L and Ag-L at pH 3, 8 and 6 respectively, because of the formation of the anionic form of the reagent, which can easily react with ions to form complexes.

Effect of reagent volume: Various volume of HL (0.5-4ml)of $1x10^{-3}$M were added to a fixed amount of Pd(II), Ni(II) and Ag(I) of 10ppm (2ml). It's found enough to develop the color to its full intensity and give a minimum blank value and were considered to be optimum for the volume 3, 3 and 2ml respectively. Figure 3 show the effect of concentration HL with ions.

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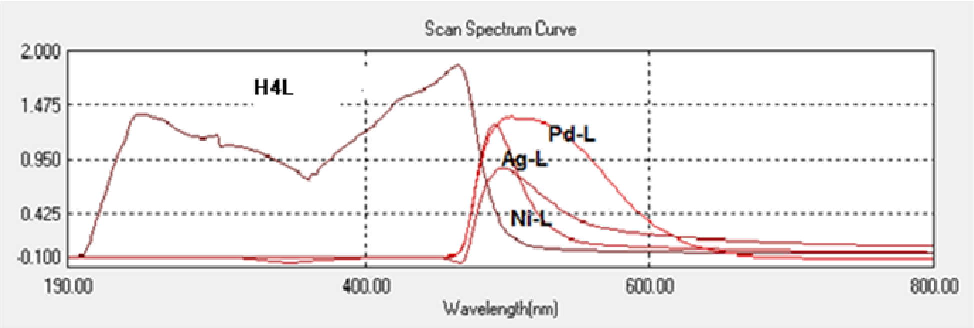

Figure 1 The absorbance λ_{max} of H_4L and their complexes.

Table 2 Analytical characteristics of metal-l complexes

Characteristic	Pd(II)	Ni(II)	Ag(I)
Absorption maxima (λ_{max}) nm	504	495	492
Bee's law range(μg/ml)	0.1-3	0.1-3	0.1-3
Molar absorptivity (L.mol^{-1}cm^{-1})	3.565x10^3	1.966x10^3	4.735x10^3
Sandell's sensitivity (μgcm^2)	0.0298	0.0298	0.0227
pH	3	8	6
Stability constant	1.406x10^{10}	2.191x10^{10}	1.2x10^7

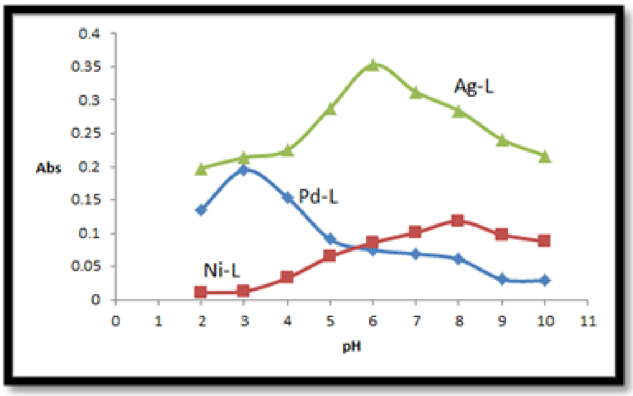

Figure 2 Effect of pH.

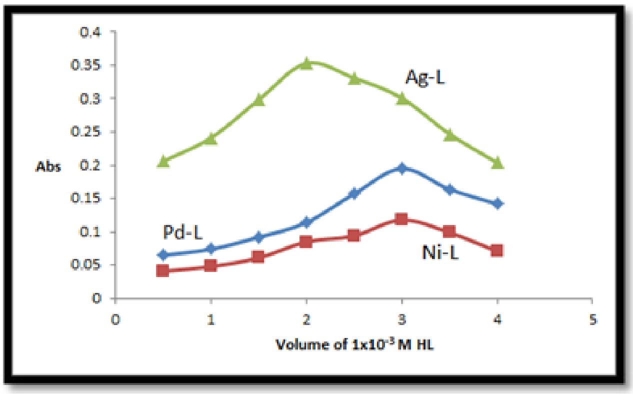

Figure 3 The effect of concentration HL on Pd(II), Ni(II) and Ag(I) ions.

Effect of time: The stability of complexes with time was showed in Figure 4, from the data obtained it was found that the highest absorbance reached at 15min and remains constant up 24hrs with respect to Pd-L, Ni-L and Ag-L respectively.

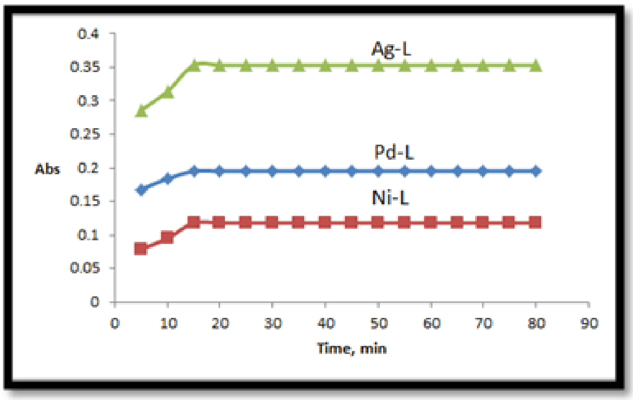

Figure 4 The effect of time.

Calibration graph: At optimum conations, a linear calibration graphs for Pd-L, Ni-L and Ag-L were obtained, (Figure 5-7) that Beers low is obeyed over the concentration range of (0.1-3ppm) Pd(II), Ni(II) and Ag(I) with R^2 equal to (0.9978), (0.9981) and (0.9982) respectively. The results of analytical performance are summarized in Table 3.

Conductivity measurements: The solubility of the complexes in dimethylsulfoxide (DMSO), ethanol and methanol permitted of the molar conductivity of 1x10^{-3} M solution at 25°C and by comparison, the electrolytic nature of complexes. The lower values of the molar conductance data listed in Table 3 indicate that the complexes are non-electrolytes.

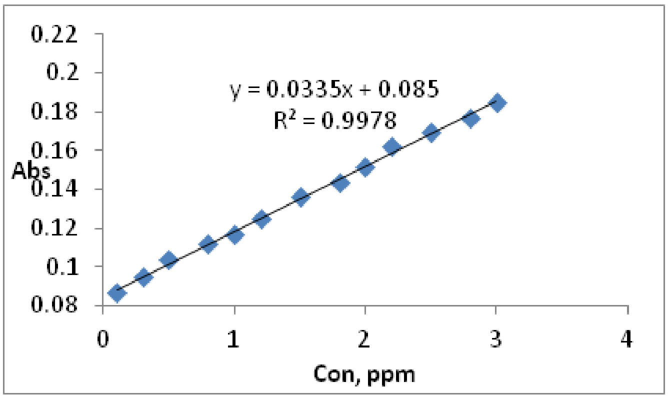

Figure 5 Calibration curve of Pd-L.

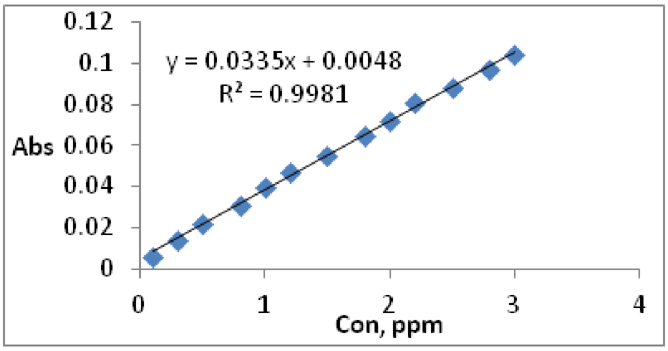

Figure 6 Calibration curve of Ni-L.

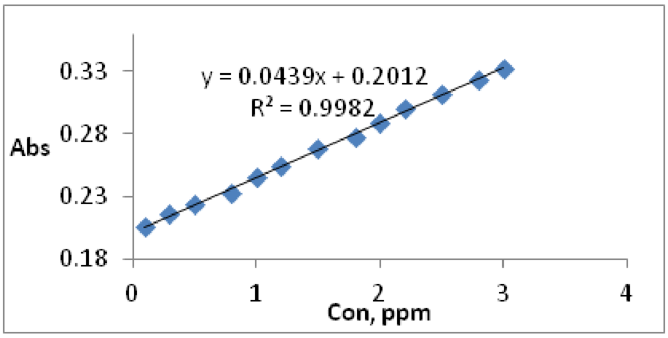

Figure 7 Calibration curve of Ag-L.

Accuracy and precision

To determine the accuracy and precision of the method, Pd(II), Ni(II) and Ag(I) were determined at three different concentrations. The results shown in Table 4 a satisfactory precision and accuracy with the proposed method.

Effect of interferences: The effect of diverse ions as interferences was studied. To test of diverse ions was determined by the general procedure, in the presence of their respective foreign ions. In the experiment, a suitable amount of the standard ions solution, coexisting ion solution and masking agent were added. The metal ions can be determined in thepresence of a 10 fold excess of cation and anion, the results are listed in Table 4. They found that large amounts of NO_3^{-1}, Cl^{-1}, CO_3^{-2} and SO_4^{-2} have a few effects of ions. It's found that positive ions interfere seriously and can make the absorbance increase or decrease. However, their interferences are masked efficiently by the addition of (0.5-2.0ml) of 0.1M of sodium nitrate for palladium and nickel and (0.5-1.5ml) of 0.1M of oxalic acid for silver.

Composition of the complexes (stoichiometery): The empirical formula of the complexes was evaluated by using a continuous variation method (Jop's method) and Mole Ratio Method.[14] It was found that complexes form 1:2 for Pd (II) and Ni(II) but 1:1 for Ag(I) (M:L), (Figure 8-13) show the Composition of the complexes. On the basic of the FTIR, stoichiomstric and conductivity data, the structures of complexes can be suggested as the following (Figure 14 & Figure 15).

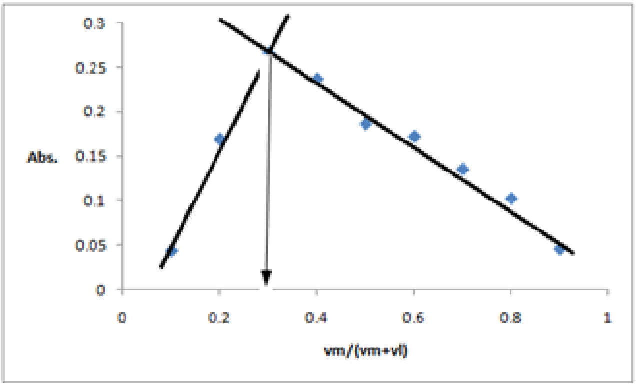

Figure 8 Job's method of the Pd-L.

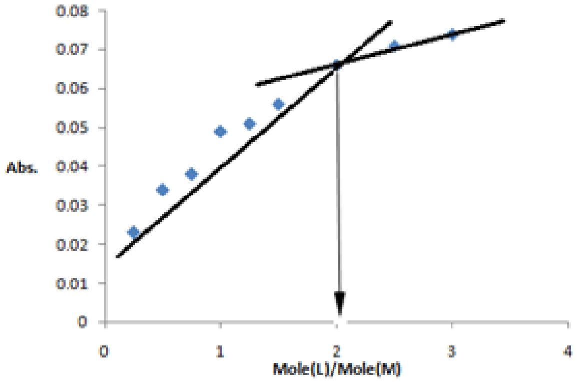

Figure 9 Mole ratio of the Pd-L.

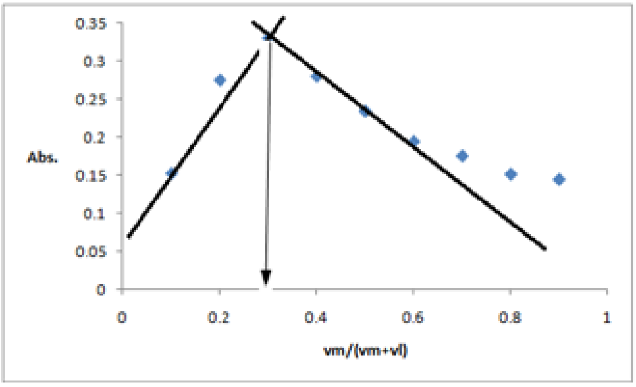

Figure 10 Job's method of the Ni-L.

Thermal analysis: TG/HDSC analyses are very useful methods for investigating the thermal decomposition of solid substances involving simple metal salts as well as for complex compounds.[15,16] The thermogram follows the decrease in sample weight with the linear increase in heat treatment temperature ($10^0C\ min^{-1}$) up to 400^0C. The decomposition occurs in at least three major detectable steps, each step does not refer in generally to single process, but rather is reflects of two or three overlapping processes and attributed to the ligand alone or companied by chlorine atoms. The aim of the thermal analysis is to obtain information concerning the thermal stability of the investigated complexes as seen in Table 5 and (Figure 16-19), to decide whether water molecules are inside or outside the coordination sphere. All the complexes show three-stage mass loss in their TG/HDSC curves.

Table 3 Conductivity values of complexes

Complexes	Molar conductivity S mol^{-1} cm^2 DMSO	Molar conductivity S mol^{-1} cm^2 Ethanol	Molar conductivity S mol^{-1} cm^2 Methanol
Pd-L	9.6	11.2	13.4
Ni-L	7.3	8.7	11.8
Ag-L	10.9	12.6	14.5

Table 4 Accuracy and precision values of complexes

Ion	Con. ppm	%R.S.D*	%Error	D.L	%Recovery
Pd(II)	0.1	0.93136	0.7604	0.00279	99.2395
	0.5	0.93786	0.79491	0.01406	99.2050
Ni(II)	1.0	0.96817	0.39525	0.02904	99.6047
	1.5	0.93324	0.60240	0.04199	99.3975
Ag(I)	0.1	0.94143	0.16155	0.002374	99.8384
	0.5	0.96153	0.29673	0.014423	99.703

*for six determinations

Table 5 Effect of foreign ions

Foreign Ions	Formula	Amount added µg/ml	Interferences with Pd	Interferences with Ni	Interferences with Ag
Cu^{2+}	$CuCl_2.2H_2O$	100	6.66	4.23	10.48
Pb^{2+}	$PdCl_2$	100	-1.02	-2.54	-1.19
Co^{2+}	$CoCl_2.6H_2O$	100	1.53	5.93	17.56
Fe^{3+}	$FeCl_3$	100	8.2	10.16	16.14
Mn^{2+}	$MnCl_2$	100	12.3	22.88	-2.26
Hg^{2+}	$HgCl_2$	100	58.97	14.4	-7.93
V^{5+}	NH_4VO_3	100	10.25	19.49	18.9
Cl^-	KCl	100	1.33	1.07	-2.6
CO_3^{2-}	Na_2CO_3	100	1.02	2.37	4. 89-
Br^-	NaBr	100	-1.5	1.93	3.14
$SO4^{2-}$	Na_2SO_4	100	-1.32	1.62	1.41
I^-	KI	100	-0.79	-1.23	-1.39

For H_4L $C_{25}H_{16}N_4O_9$ from the TG curve, it appears that the sample decomposes in two stages. The first stage decomposition occurs at 207.1^0C with a mass loss of 2.0% and the second decomposition at 352.8^0C with a 48% mass loss. For palladium complexes ($C_{50}H_{34}N_8O_{18}Pd$), the data obtained support the proposed structure and indicate that Pd(II) complex undergo three step degradation reaction. The first step occurs at maximum peak lying in 85.6^0C, corresponding to the loss of 2% the weight loss associated with this step agrees quite well with the loss one terminal methyl groups in 4-aminoantipyrine moiety. The second step occurs at T_{max} 150.7^0C, corresponding to the loss of 3% and it referred to loss of chlorine atom. The third decomposition step occurs at T_{max} 354.6^0C corresponding to the loss of 25% referred to a single process, but it's reflective of two or three overlapping processes and attributed to loss of the 4-aminoantipyrine and moieties. The residual is in agreement with Pd metal. For Ni complex ($C_{50}H_{34}N_8NiO_{18}$), the data obtained support the proposed structure and indicate that Ni(II) complex undergo three step degradation reaction. The first step occurs at maximum peak lying in 157.7^0C, corresponding to the loss of 2% the weight loss associated with this step agrees quite well with the loss of one water molecule. The second step occurs at T_{max} 189.2^0C, corresponding to the loss of 6% and it referred to loss two terminal methyl groups in 4-aminoantipyrine moiety The third decomposition step occurs at T_{max} (358.3^0C) corresponding to the loss of 35% referred to a single

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process, but it's reflective of two or three overlapping processes and attributed to loss of the 4- aminoantipyrine and moieties. For Ag complex ($C_{25}H_{16}AgN_5O_{12}$), a mass loss occurred within the temperature 115.6^{0}C corresponding to the loss of 1% for one molecule of water the temperature 179.7^{0}C a loss of 5.33%, corresponding to a loss of one NO_3 molecule at the end of the thermogram at higher temperature 356.3^{0}C.[17]

Table 6 Thermal Analysis

Compound	**First stage**			**Second stage**			**Third stage**		
	Temperature ^{0}C	M. wt	Weight loss %	Temperature ^{0}C	M. wt	Weight loss %	Temperature ^{0}C	M. wt	Weight loss %
$C_{25}H_{16}N_4O_9$	207.1	506.09	2	352.8	268.538	48	-		-
$(C_{25}H_{16}N_4O_9)_2Pd$	85.6	1126.08	2	150.7	1110.865	3	354.6	852.17	25
$(C_{25}H_{16}N_4O_9)_2Ni$	157.7	1169.63	2	189.2	1121.89	6	358.3	771.796	35
$(C_{25}H_{16}N_5O_{12})Ag$	115.6	681.726	1	179.7	649.697	5.33	356.3	466.684	32

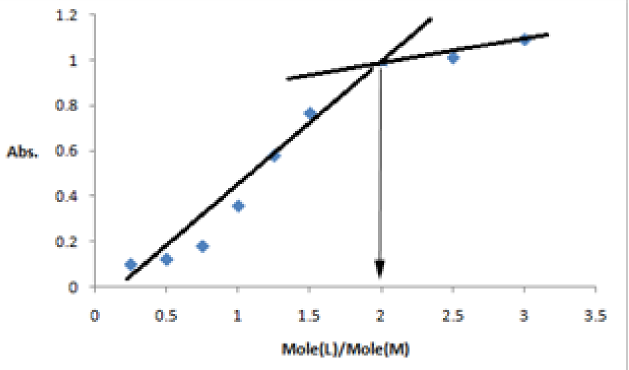

Figure 11 Mole ratio of the Ni-L.

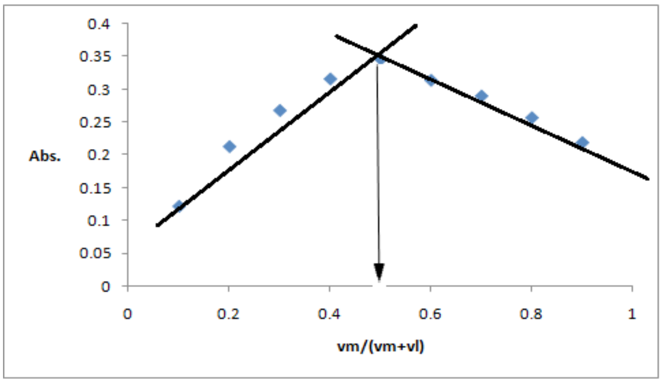

Figure 12 Job's method of the Ag-L.

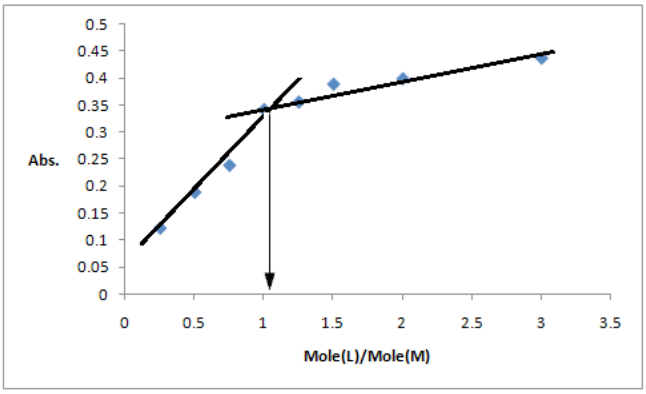

Figure 13 Mole ratio of the Ag-L.

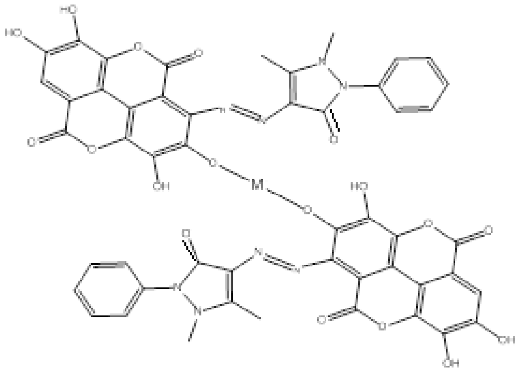

Figure14 The proposed structural formula of metal complexes (M=Pd(II) and Ni(II)).

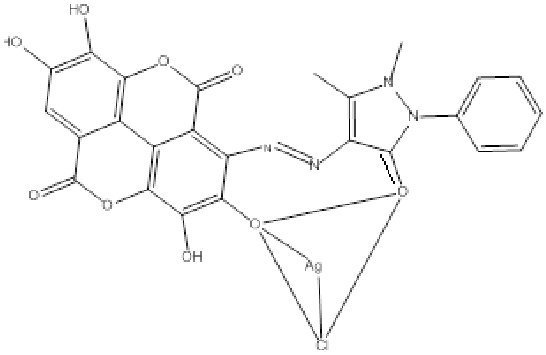

Figure 15 The proposed structural formula of Ag metal complex.

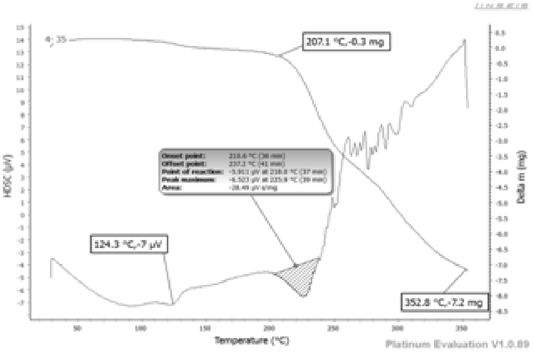

Figure 16 TG/HDSC thermogram of HL.

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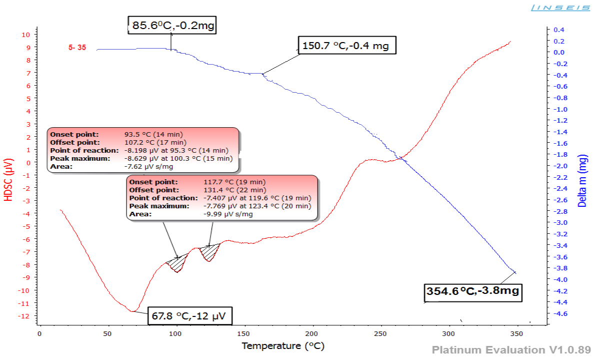

Figure 17 TG/HDSC thermogram of Pd-L.

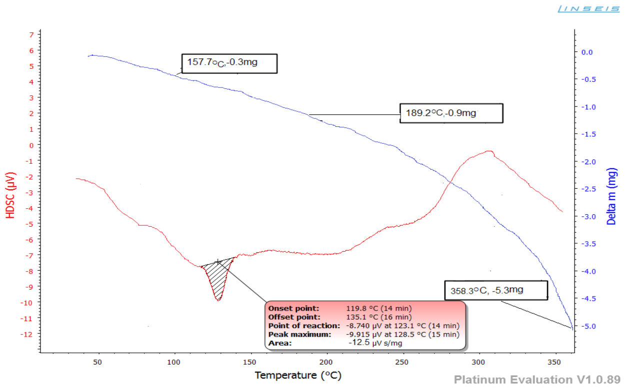

Figure 18 TG/HDSC thermogram of Ni-L.

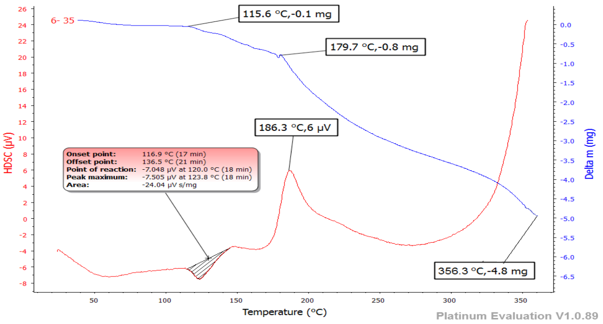

Figure 19 TG/HDSC thermogram of Ag-L.

Applications

To determination of ions determined according to the spectrophotometric method and atomic absorption in Al-Kufa and Al-Kfel rivers, we use the following methods.[18] For Pd(II): 500ml of water sample was concentrated to about 10ml by heating on a hot plate. 10ml of nitric acid and 5ml of 30% hydrogen peroxide were added in this solution. The mixture was heated on a hot plate and evaporated to near dryness. The residue was dissolved in 10ml of 10% HCl and transferred into a calibrated flask (100ml).For Ni(II):The water sample (500ml) was collected in a clean container and evaporated to about 25ml. Then 5ml of H_2O_2 was added and evaporated to dryness. It was then dissolved in 20ml of water and filtered to remove insoluble substances. The filtered was collected in 100ml volumetric flask quantitatively to the mark with distilled water. For Ag(I): The water was filtered through filter paper no. 41. The pH of filtered sample was adjusted to 2 with 1:1 nitric acid solution. The results obtained are given in Table 7.

Table 7 Determination of Pd(II) Ni(II)and Ag(I) in water

Samples	Pd(II)		Ni(II)		Ag(I)	
	atomic absorption µg.ml^{-1}	Spectro. method ppm	atomic absorption µg.ml^{-1}	Spectro. method ppm	atomic absorption µg.ml^{-1}	Spectro. method ppm
Al-Kufa	0.0156	0.0158	0.0479	0.0477	0.0146	0.0145
Al-Kfel rivers	0.0027	0.0025	0.0403	0.0401	0.0075	0.0074

Acknowledgements

None.

Conflict of interest

The author declares that there is no conflict of interest.

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