Synthesis Structural and Spectroscopic Study for Pd (II) complexes with 4-aminoantipyrine and Sodium pyrophosphate.

Sajid M. Lateef *   **Sattar R. Majeed****   **Hanaa A. Mohammed****

* College of Education for Pure Science (Ibn Al-Haitham), University of Baghdad, Baghdad, Iraq
** College of Science, University of Anbar, Ramadi, Anbar, Iraq

A R T I C L E   I N F O

A B S T R A C T

Pd (II) complexes were synthesized from 4-Aminoantipyrine (4-AAP) as primary ligand, and disodium pyrophosphato as secondary ligands using metal: ligand in (1:1) mole ratio with molecular formula [Pd(4-AAP)Cl$_2$],[Pd(4-AAP) (Pyph)]. These complexes were characterized by elemental microanalysis (C.H.N), (A.A), [I.R, (U.V–Vis), mass spectroscopy], along with molar conductivity, Chloride contents and melting point measurements. The ligands (4-AAP) and (Pyph) gave square planar geometrical formula with Pd (II). Rapid, simple, sensitive and validated spectrophotometric method has been described for the determination of palladium (II) using 4-aminoantipyrine. The complex product was quantitatively measured at 363nm and the reaction conditions were studied and optimized.

1-Introduction

Antipyrine and its derivatives are widely used as analytical agents.4-amino antipyrine mainly used to develop colour in the assay of serum alkaline phosphates. Antipyrine and its derivatives such as 4-amino antipyrine and 4-dimethylamino antipyrine are known to form stable complexes with a variety of lanthanide salts [1] . A literature survey on antipyrine reveals that the 4-poritions of antipyrine is amenable to a variety of organic reactions such as the Friedcl-Craft's acylation and Mannich reaction therefore it should be possible to introduce a suitable functional group at 4-porition of the antipyrine ring and make a potentia1 new multidentate ligand for metal ions [2] . Rajasekar *et a1* [3]. Isolated new complexes of lanthanide nitrate with N, N-diethylrntipyrine-4-carboxamidc (DEAP), and characterized by chemical analysis and various physical methods such as electrolytic conductance, infrared and ^{13}C NMR spectral data. Electrolytic. In 2012 Rajasekar his Associates [4].

Were prepared the mixed ligand complexes of Cu (II), Zn (II) and Hg (II) using 4- aminoantipyrine and thiocynate ions with microwave irradiation. The prepared complexes were characterized by elemental analysis, conductance measurement, UV-Visible, FT-IR and NMR spectral

2- Experimental

2.1 Materials and Instrumentation

Palladium chloride, 4-Aminoantipyrine, TetraSodiumpyrophosphate, DMSO and ethanol were supplied by Aldrich company.

Melting points of the prepared complexes were measured with an electro thermal (Stuart melting point apparatus). Infrared spectra were performed using a Shimadzu (FT–IR)–8400S, The electronic spectra of the compounds were recorded by using double-beam (U.V-Vis) spectrophotometer type U.V 160A (Shimadzu), Electrical conductivity measurements of the complexes were recorder at (25ºC) for (10^{-3} mole.L^{-1}) solution of the samples in DMSO by using (conductivity meter, Jew wary, model 4070), The Chloride contents were determined using (686-Titro processor-665. Dosimat Matron Swiss), Elemental analysis recorder by using Euro Vector, model EA 3000 single V.3.Osinglein, Mass spectra were recorder by using the device GC-

————— * Corresponding author at: College of Science, University of Anbar, Ramadi, Anbar, Iraq
.E-mail address: Hanaa Alkubasi@gmail.com

mass QP SO A: shima (170 ev) and Spectrophotometer measurements of the absorbance complexes were recorder by using UV-1100.

2.2. Synthesis of Complexes
2.2.1. Synthesis of [Pd (4-AAP) Cl$_2$] Complex.

1.0 mmol (0.177g) of PdCl$_2$, dissolved in 30 mL ethanol, was mixed with 1.0 mmol (0.203g) of 4-Aminoantipyrine, dissolved in 20 mL ethanol. The mixture was adjusted to pH 3.5 and stirred in the reaction flask for 4 hours. The complex was precipitated, filtered off and washed thoroughly with ethanol, then with diethyl ether.

2.2.2. Synthesis of [Pd (4-AAP) (pyph)] complex.

1.0 mmol (0.177 g) of PdCl$_2$, dissolved in 30 mL ethanol, was mixed with 1.0 mmol (0.203 g) of 4-Aminoantipyrine, dissolved in 30 mL ethanol, while stirring. 1.0 mmol (0.265 g) of Na$_4$P$_2$O$_7$ was then added an the mixture was adjusted to pH 3.5 and reflexed for 4 hours under constant stirring. The complex was precipitated and separated by filtration. The filtered complex was washed thoroughly with ethanol, then with diethyl ether.

2.3. Preparation of standard solutions

A standard 1.0×10^{-3} M Palladium (II) solution was prepared by dissolving of 0.0089g of PdCl$_2$ in 50 mL ethanol. A standard solution of 4-AAP (1.0×10^{-3}) M was prepared by dissolving 0.01g of 4-AAP in 50mL ethanol. A series of phosphate buffer solution covering the pH range 3.0 to 11.0 was prepared (0.1M with respect to potassium dihydrogen phosphate)with either 1M phosphoric acid and 0.1M sodium hydroxide to the desired pH.

3. Results and Discussion
3.1. Characterization of two Palladium (II) Complexes.
(3.1.1) Solubility

Solubility of two prepared complexes was tested in different solvents. The results were listed in **table (3-1)**.

(3.1.2) Elemental microanalysis and some physical properties

Some physical properties were listed in **Table (3-2)**. Elemental microanalysis (C.H.N), metal and chloride analysis are in a good agreement with calculated values, **Table (3-3)**.

(3.1.3) Molar conductance complexes.

The molar conductance in DMSO solvent of complexes [Pd (4-AAP) Cl$_2$] and [Pd(4-AAP)(Pyph)]lie in the (6.21-1.90) S.cm^2.mole^{-1}range, indicating their non-electrolytic behavior [5].

(3.1.4) FT-IR spectral data of Pd (II) Complexes.

IR spectral data of two complexes [Pd (4-AAP)Cl$_2$) **Fig.(3-1)** summarized in **table (3-4)**. The I.R spectra of two prepared complexes were compared with that of free ligand .The detected band at (3433) cm^{-1} and (3325) cm^{-1} which was assigned to the stretching frequency of (4-AAP) groups (υ_{asy-} NH$_2$) and (υ_{sy-} NH$_2$) of the free ligand, this band was shifted to lower or higher frequency at range (3479, 3460) cm^{-1} and (3421,3406) cm^{-1} in spectra of tow prepared complexes; this shift refers to coordination of NH$_2$ group via N atom with metal ions Pd(II). The coordination of the NH$_2$ group has been also confirmed from the shifts of its deformation vibration found at (1647) cm^{-1} in the spectrum of the ligand which was shifted to lower frequency in the spectrum of complexes to (1616,1585) cm^{-1}. The band at (1678) cm^{-1} stretching vibration which refers to $\upsilon_{C=O}$ for 4-AAP ring of free ligand, was shifted to lower frequency at (1635, 1616) cm^{-1} in the spectra of complexes, showing that the coordination between oxygen atom of this group(C=O) and metal ions was happened [6,7]. In spectra of complexes [Pd(4-AAP)(Pyph)] the band due υ(P-O-P) appeared at (1118, 1149) cm^{-1} and (910) cm^{-1} in spectrum of the ligand .was shifted to higher frequency at (1161) cm^{-1} complex respectively . But the band at (910) cm^{-1} which refers to υP=O, was found at the same position with change in shape and intensity in spectra of complex. The shifted in band of υ P-O-P and the change in intensity and shape of band (υP=O) was attributed to coordination of pyrophosphate ion with metal ions Pd(II), via two O atoms [8,9]. The I.R spectra of Pd(II) complexes exhibited

new two bands which are not present in the spectrum of the free ligand, these bands are located at range (540, 590) cm^{-1} and at (482,470) cm^{-1} which are attributed to υ(M-N) and υ(M-O) respectively [10,11]. Other bands in the IR. Spectra of complexes were listed in **Table (3-4).**

(3.1.5) U.V-Vis Spectral data of Pd (II) complexes.

The u.v-vis spectral data of Pd (II) complex [Pd(4-AAP)Cl$_2$] **Fig.(3-2)** and [Pd(4-AAP)(pyph)] were summarized in **Table(3-5)** ,.in each case the spectrum showed intense absorption peak in the u.v region at (270) nm(37037) cm^{-1} (ε_{max}=1633)m^{-1}.cm^{-1} and (270)nm (37037)cm^{-1}(ε_{max}=1338)m^{-1}.cm^{-1}, for [Pd(4-AAP)Cl$_2$and [Pd(4-AAP)(pyph)] complexes respectively assigned to ($\pi \rightarrow \pi^*$) of intra-ligand electronic transition, which was shifted to lower frequency when it comparison with that of free ligand (4-AAP) at (280)nm, this shifting confirm the coordination of the ligand (4-AAP) with central metal ion Pd(II). Also the peak at (301) nm (33223) cm^{-1}(1524) m^{-1}.cm^{-1} in spectrum of [Pd(4-AAP)Cl$_2$] complex, refers to ($\pi \rightarrow \pi^*$) of intra-ligand electronic transition. The absorption peaks at (360) nm (27778) cm^{-1} (ε_{max}=432) m^{-1}.cm^{-1} and (412) nm(24272)cm^{-1} (415)m^{-1}.cm^{-1} in spectra of [Pd(4-AAP)Cl$_2$]and [Pd(4-AAP)(pyph)] complexes, were attributed to MLCT electronic transition [12] .

Pd(II) complexes [Pd(4-AAP)Cl$_2$]and [Pd(4-AAP)(pyph)] showed peak at (470)nm (21277)cm^{-1} (ε_{max}= 208) m^{-1}.cm^{-1}and (508)nm (19685) cm^{-1} (ε_{max}=263) m^{-1}.cm^{-1} respectively, wich can be assigned to spin-allowed (d-d) electronic transition type (^{1}A$_1$g $\rightarrow^1$Eg),   while spectra of [Pd(4-AAP)Cl2] and [Pd(4-AAP)(pyph)] complexes displayed weak absorption peak at (563)nm (17762) cm^{-1} (ε_{max}=64) m^{-1}.cm^{-1} and (612)nm (16340) cm^{-1}(ε_{max}=214) m^{-1}.cm^{-1} respectively, which can be attributed to spin-allowed (d-d) electronic transition type(^{1}A$_1$g$\rightarrow^3$A$_2$g).At last spectra of [Pd(4-AAP)Cl$_2$] complex showed weak absorption peak at (778)nm (12853)cm^{-1} (ε_{max}=22)m^{-1}.cm^{-1}, which can be attributed to spin-forbidden (d-d) electronic transition type(A$_1$g-^{3}A$_2$g). All (d-d) electronic transitions for Pd(II) complexes Pd(4-AAP)Cl$_2$] and [Pd(4AAP)(pyph)] suggest an square planner geometry around Pd(II) central ion (4d^8- configuration) [12] .

3.1.6. Mass spectrum of Pd(II) complexes.
Mass spectra of [Pd (4-AAP)Cl$_2$]

Mass spectrum of complex [Pd(4-AAP) Cl$_2$] is shown in **Fig.(3-3)**. Mass spectrum of this complex (molecular weight equals380.66) gave a parent peak at m/z = 380 (M$^+$).

Mass spectra of [Pd (4-AAP) (Pyph)]

Mass spectrum of complex [Pd (4-AAP) (Pyph)] Mass spectrum of this complex (molecular weight equals529.56) gave a parent peak at m/z = 529.3 (M$^+$) a peak at m/z = 429.3 assigned for the [M$^+$- C$_7$H$_3$O] , a peak at m/z = 297assigned   for the [M$^+$-(4-AAP-C11H9N2O+ NH2)-2Na], a peak at m/z = 429.3 assigned for the [M$^+$- C$_7$H$_3$O] and a peak at m/z = 204.2 assigned for the [M$^+$-[Pd(Pyph)].

3.1.7. Selection of the analytical wavelength

The analytical wavelength (λmax) of the complex [Pd(4-AAP) Cl$_2$] was selected at(363)nm which refers to MLCT [12].

3.1.8. Effect of PH

The effect of PH on the formation of Pd (II)-AAP complex in aqueous solution was investigated over a PH range 3-11. For this purpose, aliquots solutions of 2.0 mL of 1.0$\times$10^{-3} 4-AAP was pipetted into 0.5 ml of 1.0$\times$10^{-3} Pd (II) in 10 ml volumetric flasks, the flask was made to 10.0 ml by phosphate buffer solution (PH range 3-11). The absorbance of the solutions was recorded at 363nm; **Fig.(3-4)**. that the maximum absorbance and hence the maximum formation of the Pd (II)-AAP complex, was reached at PH 6. This PH is fixed in the whole study.

3.1.9. Effect of time on the stability of Pd (II)- 4-AAP Complex.

The stability of complex between AAP and Pd (II) was observed by measuring the absorbance, at the analytical wavelength (λ_{max}=363 nm) and PH 6, every five minutes over a period of 1 hours. The complex attained stable absorbance values after 30 minutes for the subsequent 24 hours. This increased the reliability of the method as well the applicability for large number of samples. The result is shown in **Fig.(3-5).**

3.1.10. Stoichiometry of Pd (II)- 4-AAP complex

Under the optimum conditions, the stoichiometry of the formation of Pd (II)-AAP complex was investigated by Job's method of continuous variation. [13, 14]

Job's method of continuous variation

The Job's method of continuous variation was applied as described by Yoe and Jones. [13,14] Keeping the sum of the molar concentration of Pd(II) and AAP constant, the ratio of both Pd(II) and AAP was varied (see experimental section) and the absorbance of the resultant mixtures were recorded at 363nm and PH 6 The maximum absorbance corresponds to the stoichiometric ratio, this was found at 1:1(Pd(II) : 4-AAP);**Fig.(3-6).**

3.1.11. Validity of the method
Determination of linear range

2.0 mL of 1.0 $\times 10^{-3}$ M 4-AAP was pipetted into each of ten 10-mL volumetric flask containing 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0 mL of Pd (II) (1.0 $\times 10^3$M). The solutions were made up to 10 mL by phosphate buffer solution of pH (6). After 30 minutes, the absorbance of the solutions were measured at 363 nm and plotted against Pd (II) concentration in μgmL^{-1}.**Figur**.**(3-7)**

3.1.12. Sensitivity of the method

The sensitivity of the method was tested by evaluation of both the limit of detection (LOD), limit of quantification (LOQ) and Sandell's sensitivity.[15, 16] The limit of detection (LOD) is the lowest amount of the investigated compound in a sample that can be detected, but not necessarily quantified with an acceptable uncertainly. LOD of a method is an important factor if quantitative measurements are to be made at concentrations close to it .[15]The LOD was calculated from the calibration graph of the Pd (II)-AAP system where the intercept (y_B) can be calculated. The following equations were used:

$$SD = \frac{\sqrt{(\Sigma[yi - \hat{y}]^2)}}{n-2}$$ (1)

$LOD_{abs} = y_B + 3SD$ (2)

LOD_{abs} was transferred to LOD value by the equation of the regression line.

LOD_{abs}= slope (LOD) + y_B (3)

Where yi and y$^{\wedge}$ are the measured absorbance and the absorbance from the regression equation; respectively. SD is the standard deviation of absorbance from the regression line (equation**1**), LOD_{abs} is the limit of detection of the absorbance, y_B is the intercept of the calibration line and LOD is the detection limit expressed in concentration unit.

The limit of quantification (LOQ) is the lowest concentration of compound that can be measured in a sample matrix at an acceptable level of accuracy and precision. The LOQ is generally useful parameter than LOD. The LOQ is relevant only in trace analytical methods when measurements are being at concentrations close to that limit. The LOQ is always higher than the LOD and is often taken as fixed multiple of the LOD (usually triple times higher than LOD value). The LOQ is determined by the same way as LOD:

$LOQ = yB + 10SD$ (4)

 or calculated directly from the LOD: [15]

$LOQ = 3.3 \times LOD$ (5)

Sensitivity of the proposed methods is also determined by calculating Sandell's sensitivity (μg/cm^2/0.001 Abs unit), which can be defined as smallest weight of substance that can be detected in column of unit cross section. [16] The values of LOD, LOQ and Sandell's sensitivity are given in **Table(3-6)**.

3.1.13. Accuracy and precision

Accuracy is the main requirement of the determination methods. It can be described as the closeness of agreement between the value that adopted, either as a conventional, true or accepted reference value and the value found. The accuracy in this study is represented and determined by recovery studies. Accuracy of the method is indicated by the closeness of the 100% recovery value [15,16]. The % recovery ((found/taken)×100) and the percent error (%Er = (found-taken/taken)×100) were evaluated to indicate the accuracy of the proposed method. On the other hand, precision describes as the degree of agreement among individual test results when the procedure is applied repeatedly to multiple samplings of homogeneous sample. [16]The repeatability (intraday) and reproducibility precision (inter-day) were applied to the

proposed method to indicate the precision. Repeatability were performed by analyzing four synthetic Pd(II) samples whose concentrations cover the linear range of the method, each was repeatedly measured 6 times in the same day (intra-day). The reproducibility is tested by the same way as the repeatability but the measurements were performed over two weeks (inter-day) [15]. All the results are reported as the RSD% values as indication of the precision are given in **Table(3-7)**.

Method analytical features.

The brown color of Pd (II)-AAP complex obeyed Beer's law in the concentration range 1.06-9.54 μg mL^{-1} with a molar absorptivity value of $1.55x10^3$ L mol^{-1} cm^{-1} at 363 nm. The linearity is indicated from the high correlation coefficient (R^2 =0.998). The method is sensitive as indicated from the low values of LOD (0.034 $\mu g mL^{-1}$), LOQ (0.114 $\mu g mL^{-1}$) and Sandell's sensitivity (0.0305μg. cm^{-2}). The proposed method showed high accuracy as indicated by the percentage recovery (100%-100.95%) and %Er (0.0005%-0.9599%) values. The precision of the method was found to be also high as indicated from the very small values of the RSD% (0.2 -2.12%) indicating reasonable repeatability and reproducibility of the proposed method. **Table** (**3-7**). The method   is comparable with the other reported methods; **Table(3-8).**

5-Conclutions

1.According to the characterization data for new complexes Pd(II) with 4-Aminoantipyrine by FT- IR, U.V-Vis, atomic absorption, molar conductivity, elemental microanalysis, chloride content along with melting point, we found that:

1-The ligand 4-AAP and disodiumpyrophosphate behave as bidentate ligand on coordination with Pd(II) ion.

2-The square planar geometrical structure was suggested for prepared complexes based on the characterization data using all previous techniques.

3. The results obtained from the research showed that the 4-Aminoantipyrine detector can be used in spectral analysis to estimate the micrograms of palladium ions. As it have complexes with (colored solutions or colored sediments). These complexes have super absorbent peaks in which these ions can

be quantitatively for concentrations within the calibration curve for each ion.

Table (3-1) Solubility of the complexes Pd (II) in different solvents.

NO	compounds	H2O	DMSO	DMF	CH3OH	C2H5OH
1	[Pd(4-AAP)Cl2]	÷	+	+	+	+
2	[Pd(4-AAP)(pyph)]	÷	+	+	+	+

Table (3-2) Some physical properties of Pd (II) Complexes.

Empirical formula	Microanalysis found, (Calc.) %			A.A	Cl
	C	H	N		
1 [Pd(AAP)Cl2]	34.70	3.42	11.04	27.95	18.65
	33.62	4.10	12.13	28.1	17.5
2 [Pd(AAP)(PyPh)]	24.93	2.45	7.93	20.09	
	23.72	2.33	6.90	19.55	

Table (3-3) Micro elemental analysis of Pd (II) complexes

Complexes	M-wt - g/mol	Yield %	Colour	m.p. ºC
[Pd(AAP)Cl$_2$]	380.66	80	Brown	Over 250
[Pd(AAP)(Pyph)]	529.56	79	Dark brown	232-235

Table (3-4) Important IR spectral data of complexes

Compound	$\upsilon_{asy.}NH_2$	$\upsilon_{sy.}NH_2$	υ C=O	δN-H	υP-O-P	υP=O	υM-N	υM-O
[Pd(AAP)Cl$_2$]	3479	3421	1635	1616			540	482
[Pd(AAP)(Pyph)]	3460	3406	1616	1585	1161	910	590	470

Table (3-5) Electronic spectral data of ligand (4-AAP) and its Pd (II) complexes

No.	Compound	Wave number	ε_{max} molar^{-1} cm^{-1}	Assignment	Suggested structur

Compound		nm	cm⁻¹	ε	Assignment	Geometry
1	4-AAP	280	35714	2413	$(\pi \rightarrow \pi^*)$	
2	[Pd(4-AAP)Cl₂]	270	37037	1633	Intra-ligand	Sq. Plan.
		301	33223	1524	Intra-ligand	
		360	27778	432	MLCT	
		470	21277	208	$^1A_{1g} \rightarrow {}^1E_{1g}$	
		563	17762	64	$^1A_{1g} \rightarrow {}^1A_2g$	
		778	12853	22	$^1A_{1g} \rightarrow {}^3A_2g$	
		382	26178	394	MLCT	
		419	23866	263	$^1A_{1g} \rightarrow {}^1Eg$	
		783	12771	29	$(^1A_{1g} \rightarrow {}^3A_2g)$	
3	[Pd(4-AAP)(PyPh)]	270	37037	1338	Intra-ligand	Sq. Plan.
		412	24272	415	MLCT	
		508	19685	262	$(^1A_{1g} \rightarrow {}^1Eg)$	
		612	16340	214	$(^1A_{1g} \rightarrow {}^1A_2g)$	

Table (3-6) Analytical parameters (Using AAP-Reagent).

Parameter	Value
λ_{max} nm	363
Beer's Law Limit (μg mL^{-1})	1.06 -9.54
Molar Absorptivity (Lmol^{-1}cm^{-1})	1.55 x10³
Limit of Detection (μg mL^{-1})	0.034
Limit of Quantification (μg mL^{-1})	0.114
pH	6
Slope (mLμg^{-1}cm^{-1})	0.0327
Intercept	0.0188
Correlation Coefficient (R²)	0.998
Sandell's Sensitivity (μg cm^{-2})	0.0305
% RSD	0.2-2.12
% Recovery	100%-100.95
% Er	0.0005%-0.959

Table (3-7) Evalution of accuracy and precision(intra-and inter-day) of the spectrophotometric determination Palladium with 4-AAP.

No	Taken (μg)		Intra-day					Inter-day			
		Found (μg)	Recov (%)	SD	%RSD	%Er	Found (μg)	Recov (%)	SD	%RSD	%Er
1	1.06	1.060005	00.005	0.022507	2.123295	0.000505	1.07017	100.95	0.0051	0.48524	0.959943
2	2.12	2.120014	100.006	0.014607	0.689009	0.000642	2.120068	100.03	0.0080	0.38135	0.003212
3	3.18	3.18	100	0.010821	0.340292	0	3.180001	100	0.0082	0.26083	2.36E-05
4	4.24	4.24002	100.005	0.00621	0.146466	0.000465	4.240027	100.00	0.0084	0.20035	0.000636

59

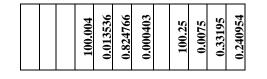|		
100.004	100.25	
0.013535	0.0075	
0.824796	0.33195	
0.00000	0.240954	

Table (3-8) Comparison of the 4-AAP method for the determination of Pd (II) with other reported methods.

Parameter	This method	[17] ref	[18] ref	[19] ref	[20] ref
λmax nm	363	315	438	684	352
Beer's Law Limit (µg mL^{-1})	1.06 -9.54	2.79-8.36	0.2-2.2	0.1-2.8	0.7 -16.5
Molar Absorptivity (Lmol^{-1}cm^{-1})	1.55 x10^3	2.26×10^4		6.70 x10^3	1.22 x 10^4
Limit of Detection (µg mL^{-1})	0.034	0.396	0.07	0.096	0.07
Limit of Quantificatio n (µg mL^{-1})	0.114	1.2	0.2	0.160	0.21
PH	6	4	8	9-10	2.6
Slope (mLµg^{-1}cm^{-1})	0.0327	-	-	0.063	-
Intercept	0.0188	-	-	0.0058	-
Correlation Coefficient (R2)	0.9974	-	0.9997	0.996	-
Sandell's Sensitivity (µg cm^{-2})	0.0305	0.007215	-	0.0160	0.002
% RSD	0.2-2.12	-	-	1.40	-

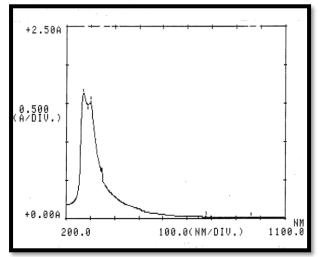**Fig. (3-1) FT-IR spectrum of [Pd (4-AAP) Cl2]**

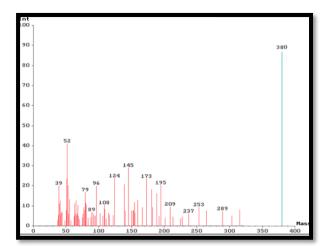**Fig. (3-2) Electronic spectrum of [Pd (4-AAP)Cl2]**

Fig.(3-3) Mass spectrum of [Pd (4-AAP)Cl2]
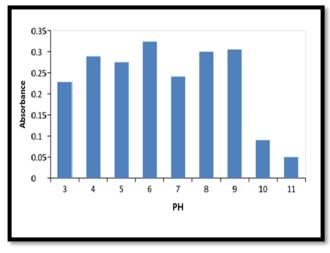
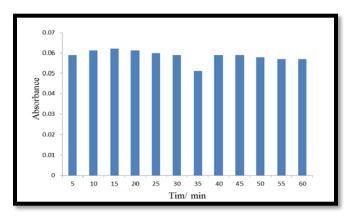**Fig (3-4): Effect of pH on the formation of Palladium (II)-4-AAP complex**

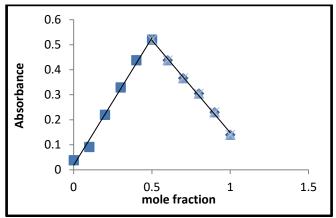**Fig. (3-5): Effect of time of the formation of Pd-AAP complex**

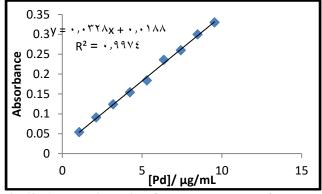**Fig. (3-6): Job's plot of complex Pd (II)- 4-AAP at PH= 6**

Fig.(3-7): Relationship of the absorbance of complex between Pd (II) and 4-AAP by increasing concentration of Pd (II) in excess 4-AAP solution

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تخليق ودراسة طيفية لمعقدات البلاديوم الثنائي مع 4-امينو انتي بايرين والصوديوم بايروفوسفيت

ساجد محمود لطيف   ستار رجب مجيد   هناء عبدالرحمن محمد

Email : Hanaa Alkubasi@gmail.com

الخلاصة

تم تحضير المعقدات الصلبة للبلاديوم الثنائي مع 4- امينو انتي بايرين وكذلك حضرت المعقدات مختلطة الليكاند 4-امينو انتي بايرين بصفته ليكاند اساسي مع ايون الصوديوم بايرو فوسفيتو بصفته ليكاند الثانوي مع ايون البلاديوم الثنائي باستخدام نسبة (1:1) فلز-ليكاند. شخصت المعقدات المحضرة بواسطة الطرق الطيفية (التحليل الدقيق للعناصر ,الامتصاص الذري,طيف الاشعة تحت الحمراء , طيف الاشعة فوق البنفسجية-المرئية),طيف الكتلة,التوصيلية المولارية,ومحتوى الكلور . واستنادا على النتائج المستحصل عليه اقترحنا شكل مربع مستوي للمعقدات البلاديوم الثنائي. وقدمت الدراسة طريقة طيفية بسيطة وحساسة وذات مصداقية تم تطبيقها لتقدير البلاديوم الثنائي باستخدام 4-امينو انتي بايرين. تم القياس الطيفي للمعقدات الناتجة عند الطول الموجي (نانوميتر) 363 وتم ضبط ودراسة امثل الظروف لتقدير الفلزات في الطرق المقترحة.