

Synthesis, characterization and molar conductivity of 1,1-cyclobutanedicarboxylate platinum(II) and palladium(II) complexes with histidine and tryptophane

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Abstract:

Four complexes of the type $[M(AA)(CBDCA)]$ (where $M = pt, pd$; $AA =$ histidine (His), tryptophan (Trp) amino acids which contain heterocyclic ring; $CBDCA = 1,1$ -cyclo-butandicarboxylate) have been prepared. The structural information of these complexes characterized by infrared, U.V-visible spectroscopy and molar conductivity measurements. Both the histidine and tryptophan ligands are bond with platinum(II) and palladium(II) in a bidentate fashion through the amino nitrogen and oxygen atom of carboxylate group. The electronic transitions appeared as d-d transitions and U.V-visible together with I.R spectra of these complexes have been given square planer geometry and C_{2V} point group. All the prepared complexes have shown a weak electrolyte in DMF solvent. The stability of complexes arrange according to the increase of the stability as follows: $[I] > [II] > [III] > [IV]$.

1. Introduction:

Complexes of amino acids with platinum(II) and palladium(II) are well known^[1-3]. The platinum(II) and palladium(II) complexes of amino acids have been of wide interest because of their biological aspects and the variety of their structural properties^[4-6]. Therefore, a great deal of efforts have been made to find platinum and palladium complexes containing amino acid ligands as potential antitumor agents to increase the activity and decrease the toxicity compared to antitumor drugs which have been used^[7-11]. As such, the majority of these complexes containing amino acids ligands that are bound to the platinum or palladium center in a unidentate fashion through the amino nitrogen atom or in a bidentate fashion through the amino nitrogen atom and carboxylate oxygen atom. Platinum (II) and palladium(II) complexes

containing amino acid ligands that are bound in a unidentate fashion through the carboxylate oxygen are rare⁽¹²⁾.

Because of platinum antitumor agents containing the bidentate ligand 1,1-cyclobutanedicarboxylate (CBDCA) tend to have very high therapeutic indexes^[7,13,14], we have chosen this ligand as a starting point for our platinum(II) and palladium(II) complexes. We report here the synthesis of four complexes of 1,1-cyclobutanedicarboxylate platinum(II) and 1,1-cyclobutanedicarboxylate palladium(II) complexes containing amino acids which contain hetro cyclic ligands such as: Histidine (His) and tryptophan (Trp) in order to estimate the bound fashions of these amino acids with central metal and some physicochemical properties.

Experimental.

1- Material and Instrumentation:

Barium chloride, 1,1 - cyclobutanedicarboxylic acid (CBDCA), *d*- histidine (His), potassium hydroxide, silver sulfate, *d*- tryptophan (Trp) were obtained from Aldrich chemical company (Milwaukee, W.I.). K_2PdCl_4 was obtained from Johnson Matthey (Seabrook, N.H.). All chemicals that were obtained from commercial suppliers were

used as received. 1,1-cyclobutanedicarboxylic acid was converted to its barium salt by the treatment with 2 equivalent of potassium hydroxide in 95% ethanol at boiling temperature and the resulting dipotassium salt was converted to barium salt by treating with $BaCl_2$ ^[15-16]. K_2PtCl_4 was prepared from platinum metal according to the literature procedures

^[17-18] The histidine and tryptophan were converted to their sodium salts by the treatment with 1 equivalent of sodium hydroxide.

Infrared spectra were measured as KBr disk on a Bye Unicam Sp-3 300S infrared spectrometer in the range 4000-200 cm^{-1} . U.V.– visible spectra were recorded on a thermospectronic Helios α v 4.60

2-Synthesis

Synthesis of iodoplatinum (II) and palladium

(II) complexes.

Complexes [(His) PtI_2] and [(Trp) PtI_2] also, [(His) PdI_2] and [(Trp) PdI_2] were prepared as bellow and according to the aprevious method^[19,20].

Synthesis of cis-[(His) PtI_2] and cis-[(His) PdI_2].

K_2PtCl_4 (0.83g , 2mmol) was dissolved in 20ml of distilled water and mixed with a solution of KI (1.76g ,10 mmol) in 25 ml of water . The reaction mixture was stirred at room temperature for 30 min and then 10ml of histidine potassium salt(0.354 g , 2mmol) was added dropwise to the stirred mixture . Stirring was continued for further 1hrs. giving a yellow precipitate. The precipitate was collected by filtration and repeatedly washed with distilled water, (yield 85%) . The [(His) PdI_2] was prepared in the similar manner and giving yield 81%.

Synthesis of cis-[(trp) PtI_2] and [(Trp) pdI_2]

Potassium iodide (0.83g , 5mmol) was added to a aqueous solution of K_2PtCl_4 (0.415g , 1mmol) , and the reaction mixture was stirred for 30min . Then the potassium salt of tryptophan (0.226 , 1mmol) in 10 ml of water was added to the resultant K_2PtCl_4 solution . After the reaction mixture was left for 1h at room temperature a yellow precipitate was obtained. The yellow solid collected by filtration and washed with distilled water (yield 87%) . The Cis-[(Trp) PdI_2] was prepared and worked as above, giving yield 85% .

Synthesis of complexe [I] : Cis – [(His) Pt (CBDCA)]

To a suspension of (0.602g , 1mmol) of cis[His PtI_2] in 25 ml of water (0.311g , 1mmol) of silver sulfate in 50 ml of water was added . The reaction mixture was stirred for 5hrs. and then the precipitated silver iodide was filtered off . An equimolar solution of barium 1,1-cyclobutanedicarboxylate [Ba (CBDCA)] equimolar solution of (10.279g , 1mmol) in 25 ml of water was

using dimethylformamide (10^{-4}M) solution at room temperature konduktoskop model E – 365B was applied using a standard conductivity cell with cell constant of 0.7821cm^{-1} Melting points were determined on a gallenkamp melting point apparatus.

dropped into filtrate of (His) Pt SO_4 and the reaction mixture was stirred for 24h. After the barium sulfate was filtered off , the filtrate was reduced to 5 ml , to which excess acetone was added until precipitate appears . The crude product was recrystallized using a mixture of water and acetone (1:1) to obtain brown crystals (yield 72%).

Synthesis of complex [II] : cis – [(Trp) Pt (CBDCA)]

To [(Trp) Pt I_2] (0.651g , 1mmol) suspended in 25 ml of water dropwise an equimolar aqueous solution of Ag_2SO_4 (0.311g , 1mmol) in 50 ml of water , and the reaction mixture was stirred for 5hrs. , after silver iodide was filtered off , The filtrate was added dropwise to a vigorously stir aqueous solution of barium 1,1- cyclobutanedicarboxylate (0.279g , 1mmol) and the reaction mixture was stirred for 24hrs. After the barium sulfate precipitate was filtered off , the resultant solution was condensed to 5 ml and then left to dry at room temperature . Then the solid product was recrystallized using a mixture of water and acetone (1:1) to obtain beige crystals of [(Trp) Pt (CBDCA)] (yield 72%).

Synthesis of complex [III] : cis – [(His) Pd (CBDCA)]

To a suspension of (1.026 g , 2mmol) of [(His) Pd I_2] in 25 ml of water was added to (0.622 g, 2 mmol) of silver sulfate in 50 ml of water . The reaction mixture was stirred for 5hrs. and then precipitated silver iodide was filtered off . An equimolar solution of barium 1,1-cyclobutanedicarboxylate (0.558g, 2 mmol) in 50 ml of water was dropped into the filterate of (His) Pd SO_4 and the reaction mixture was stirred to 24hrs. After the barium sulfate was filtered off , the filtrate was condensed to 5 ml , to which excess acetone was added to precipitate the solid product . The solid product was recrystallized using a mixture of water and acetone (1:1) to obtain brown crystals (Yield 68%).

Synthesis of complex (IV) : cis – [(Trp) Pd (CBDCA)]

This complex was prepared by the reacting of (1.124 g , 2 mmol) [(Trp) PtI₂] in 50 ml of water with aqueous solution (0.622 g , 2 mmol) silver sulfate . The reaction mixture was stirred for 5h. and then the precipitated silver iodide was filtered off . To the filtrate solution of (Trp) PdSO₄ (0.812 , 2

mmol) was added (0.558 g , 2 mmol) of barium 1,1-cyclobutanedicarboxylate and the resulting mixture was then stirred for 24hrs. After barium sulfate was filtered off , the filtrate was evaporated to dryness .

The solid yellow solid was recrystallized using a mixture of water – acetone (1:1) to obtain yellow crystals (Yield 65%).

Results and Discussion

Four complexes of 1,1-cyclobutanedicarboxylate Palatinum(II) and Palladium (II) with the amino acids that contain heterocyclic ring (histidine and tryptophan) have been prepared . Figure (1) illustrates the two amino acids (histidine and trptophan) and 1,1- cyclobutanedicarboxylate ligands which were used in this study . All complexes synthesized for this study are listed in table (1). All complexes were prepared by the same general procedure . The method included the reaction of K₂PtCl₄ salt with KI gave K₂PtI₄ which upon reaction with 1 equimolar of potassium salt of histidine or trptophan at room temperature led to the formation of pale yellow precipitate of [(His) ptI₂] or [(Trp)PtI₂] .

The equations (1-5) have shown the reactions of K₂PtCl₄ with 1 equivalent of potassium salts of histidine or trptophan .

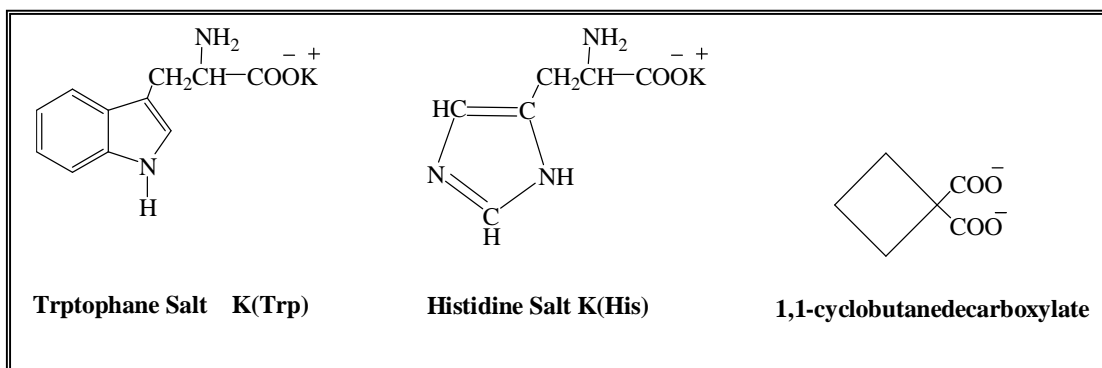
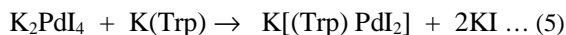
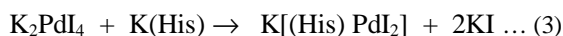
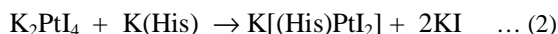
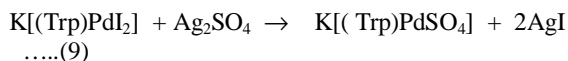
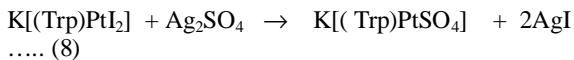
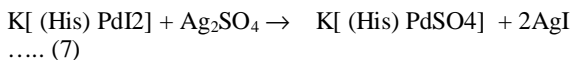
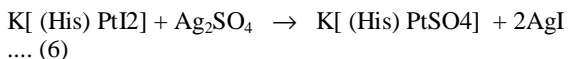


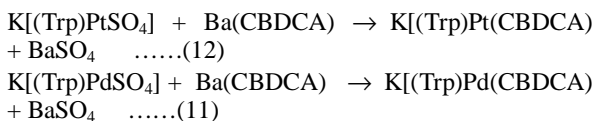
Figure (1) : salts of the ligands which used in the prepared complexes

The precipitates [(His) PtSO₄] , [(His) PdSO₄] , [(Trp)PtSO₄] and [(Trp) PdSO₄] were prepared according to the reactions(6-9) shown below.



After silver iodide or silver chloride was filtered off , the compounds of sulfato platinum(II) or palladium(II) were reacted with barium salt of 1,1-cyclobutanedicarboxylate Ba(CBDCA) to give the final complexes I,II,III and IV. The synthetic routes are shown in equations (10-13) below.





The physical properties and yields of the prepared complexes (I, II, III and IV) are given in table(1).

Table (1): Some physical properties and yield of complexes

Complex number	Complex	Physical properties		Yield
		Color	Melting point (dec °C)	
[I]	[(His) Pt (CBDCA)]	Brown	240	76%
[II]	[(Trp) Pt (CBDCA)]	Beige	232	71%
[III]	[(His) Pd (CBDCA)]	Brown	196	74%
[IV]	[(Trp) Pd (CBDCA)]	Beige	185	65%

The yield of complexes are good , this explained the stability of these complexes and demonstrate that these amino acid may be bound to platinum(II) or palladium(II) to form five membered chelate's ring, which is among stable chelates.

All the complexes are soluble in water because of their ability to form hydrogen bonding with water. the complexes are also soluble in methanol , ethanol , dimethyleformamide and dimethylsulfoxide are due to the high polarity of their anionic ligands . All the complexes decomposed in the temperature range (185-240 °C). They have been characterized by infrared and U. V.- visible spectroscopy. The infrared spectra have been recorded for each of the complexes [I]-[IV], figures (2-5) and shown in Table (2). From I.R. spectra, the amino acid coordination mode with the metal center. Figure (2-5) displays the patterns it seem that typical of amino acid which is bound in a bidentate fashion to a metal center. One of these bound fashion through the nitrogen atom of amino acid ligand appeared as one stretch band in the range of (558-565 cm⁻¹). The second bound fashion through oxygen atom of the carboxylate group appeared as one stretch band of (380-400 cm⁻¹). These vibration frequencies were corresponding with early studies which indicate to

bound platinum(II) or palladium(II) with the amino acid in (ON) mode [22,23].

The carbonyl group of the amino acids show two strong vibrational absorption frequencies bands , the first followed the ν_{asy} (C=O) band was observed in the region (1530-1590 cm⁻¹) and the other to the ν_{sy} (C=O) band in the region (1385-1430 cm⁻¹) On the other hand, the carbonyl group of (CBDCA) ligand shows two strong vibrational bands, the first ν_{asy} (C=O) ranged (1595-1620 cm⁻¹) and the second ν_{sy} (C=O) in the range (1325-1365 cm⁻¹) while (M-O) bond between CBDCA with metal center in the range (380-400 cm⁻¹) .

The shift in the carbonyl frequencies for His , Trp and (CBDCA). compared to free ligands , as shown in the table (3) , were considered the second indication to bound the His , Trp and (CBDCA) from the carboxyl oxygen atom . There are two absorption bands in the range (3005-3213 cm⁻¹) due to the (N-H) stretch and the shift in the (N-H) stretch towards low frequencies compared to free amino acids, as shown in table (3), indicate the histidine and trptophan bound with metal center through NH₂ group of amino acid.

Table(2):The important frequencies in I.R spectra in cm⁻¹ unit of the complexes.

Complex	Amino acid				(CBDCA)			
	Str* (N-H)	ν_{asy} (C=O)	ν_{sv} (C=O)	(M-N)	(M-O)	ν_{asy} (C=O)	ν_{sv} (C=O)	(M-O)
[(His) Pt (CBDCA)]	3200 - 3005	1565	1385	565	380	1600	1330	420
[(Trp) Pt (CBDCA)]	3205 - 3105	1530	1405	562	390	1596	1365	415
[(His) Pd (CBDCA)]	3213 - 3015	1570	1405	560	385	1595	1335	420
[(Trp) Pd (CBDCA)]	3215 - 3090	1590	1430	558	400	1620	1355	425

Table (3) : The shift between free ligands and complexes

Complex	[I]	[II]	[III]	[IV]
$\nu_{\text{asy}}(\text{C=O})$ of free amino acid	1751	1747	1751	1747
$\nu_{\text{asy}}(\text{C=O})$ of complex	1565	1530	1570	1590
$\Delta \nu$	186	217	181	157
$\nu_{\text{asy}}(\text{C=O})$ of (CBDCA) in complex	1600	1590	1595	1620
$\Delta \nu^*$	4	7	5	6
(N-H) of free amino acid	3247	3261	3247	3261
(N-H) of complex	3200	3205	3213	3205
$\Delta \nu^{**}$	47	56	34	56

where :

$$\Delta \nu = [\nu_{\text{asy}}(\text{C=O})]_{\text{free amino acid}} - [\nu_{\text{asy}}(\text{C=O})]_{\text{complex}} ;$$

$$\Delta \nu^* = [\nu_{\text{asy}}(\text{C=O})]_{\text{free (CBDCA)}} - [\nu_{\text{asy}}(\text{C=O})]_{\text{complex}} ;$$

$$\Delta \nu^{**} = [\nu(\text{N-H})]_{\text{free amino acid}} - [\nu(\text{N-H})]_{\text{complex}} .$$

The U. V- visible absorption spectra of the complexes [I] – [IV] have been recorded in (10^{-2} m) using methanol as solvent in the range of (200-600 nm), figures (6-9) and summarized in table (4). The complexes spectra show one electronic transition in the range (303 nm : 33003.3 cm^{-1} – 293 nm : 43129.7 cm^{-1}) due to d-d transition between unfilled 4d of palladium(II) or 5d of platinum(II) orbitals . On the other hand , although these transitions are forbidden they have a good intensities ranged ($\text{dm}^3 \text{ cm}^{-1} \text{ ma}^{-1}$) and may be due to the internal vibrations in the electronic levels or the distortion on square planner, except complex (II) that has low intensity compared to other complexes . The absorption band in the complexes are due to singlet $^1A_1 \rightarrow ^1B_2$ i.e. $dx^2 - y^2 (a_1) \rightarrow dxy (b_2)$ with ϵ values ranged ($56.8 - 251.4 \text{ dm}^3 \cdot \text{cm}^{-1} \cdot \text{mol}^{-1}$).

Table (4) : Absorption bands of U.V - visible for the complex (I)–(IV) .

Complex	Absorption bands (nm)	Absorption(cm^{-1})	E($\text{dm}^3 \text{ cm}^{-1}$)
[I]	300	33333.33	179.6
[II]	293	34129.70	56.8
[III]	303	33003.30	251.4
[IV]	299	33444.82	157.1

The molar conductivity measurements have been made for complexes (I) – (IV) in DMF solvent at (10^{-4} M). Figures (10-11) has shown that

The U.V – visible data supported the conclusion suggested from I.R data that all complexes must be in cis isomer which have C_2V point group^[26].

When the replacement of the histidine by trptophane ligand , i.e. Comparing the complexes (I) and (II) which contain platinum (II) , we observed change in the energy and intensities towards low wavelength (blue shift) and low intensity . These observation are due to the different coplanarity of these complexes from square planar , also , because the trptophan ligand has high reduced effect and crystal field effect compared to histidine ligand .

The comparison between complexes (I) and (III) , also , complexes (II) and (IV) that have the same ligands and different metals , observed red shift (high wavelength) and high intensities because the platinum metal lying in the third period of the transition metals which have high crystal field splitting energy on the d – orbital compared to the palladium metal lying in the second period of the transition metal .

electrochemical behavior for these complexes in water is high, they give high molar conductivity. Because of the solubility of these complexes in

water is high and their give high molar conductivity because of lable charge on the complexes . The molar conductivity is in the range (586 – 639 $\text{ohm}^{-1} \text{mol}^{-1}$) which is above the (118-131) $\text{ohm}^{-1} \text{cm}^{-2} \text{mol}^{-1}$ range which displays rather high molar conductivity

to consider the complexes to be 1:1 electrolyte⁽²⁴⁾ . The solutions were allowed to age for up to 24h. and no detectable increase in molar conductivity was observed in platinum (II) complexes but the opposite for palladium (II) complexes, table (5) .

Table (5) : Molar conductivity in difference time in 10^{-4} M

Complex	$\Lambda \text{ Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$	(10^{-4} M)
	Time = 0 h.	Time = 24 h.
[I]	586	586
[II]	620	621
[III]	594	605
[IV]	645	645

The change in molar conductivity of palladium (II) complexes are enough to suggest that palladium (II) complexes are unstable in their solution, which may be due to the high liability of palladium (II) complexes in solution compared to platinum (II) complexes which described in high stability especially with the ligands which contain five membered ring with metal center⁽²⁵⁾.

Generally the complexes which contain (Trp) ligand, complexes (II) and (IV) gave high molar conductivity than the complexes which contain (His) in complexes (I) and (III). In order to know the stability of the complexes [I]-[IV], we calculated the dissociate constant (Kd) and molar conductivity in zero concentration (Λ_0) by using krauss equation⁽²⁶⁾. Figures (10,13) and table (6) have shown the (Kd) and (Λ_0) of these complexes.

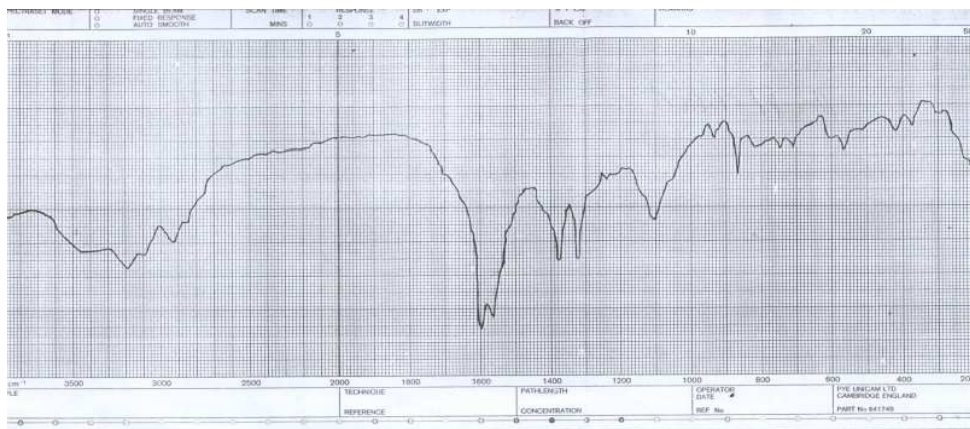
The Kd values represent the stability of these complexes which can be arranged according to the increase Kd (decrease in stability) magnitudes, as follows [IV] > [III] > [II] > [I] . The (Λ_0) arranges as follows: [I] > [II] > [III] > [IV] .

From these values we can conclude that palladium (II) complexes with (His) and (Trp) ligands are less stable than platinum (II) with same ligands due to the higher liability of palladium (II) complexes compared with platinum (II) complexes.

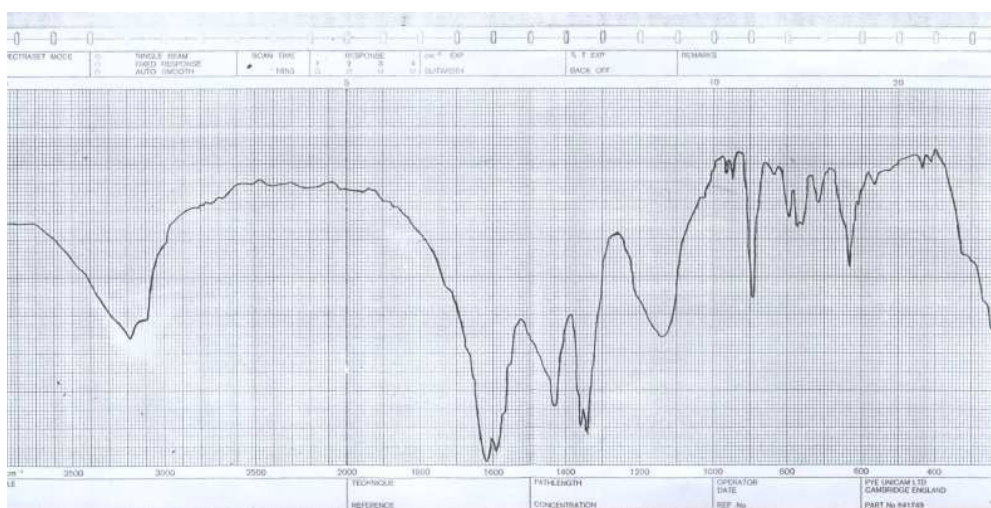
On the other hand, the platinum (II) and palladium (II) complexes with (Trp) ligands are less stable than with (His) ligand, this has been attributed to the basic behavior of (His) ligand compared to (Trp) ligand which has neutral behavior.

Table (6): Dissociation constants (Kd) and molar conductivities in zero concentration for complexes.

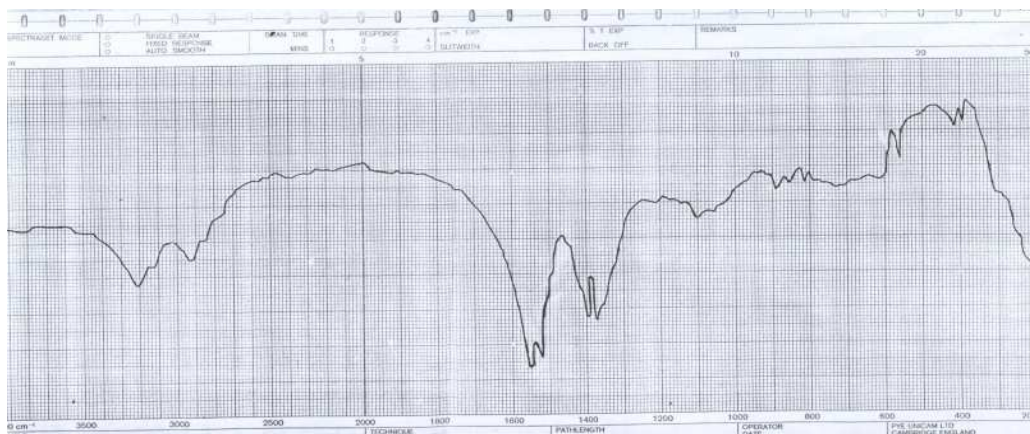
Complexes	(Λ_0) $\times(\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1})$	Kd $\times(\text{mol. cm}^{-3})$
[I]	833.33	1.2×10^{-6}
[II]	666.66	1.4×10^{-6}
[III]	454.54	4×10^{-6}
[IV]	434.70	6.4×10^{-6}



Figure(2):The infrared spectra of the complexes [I]



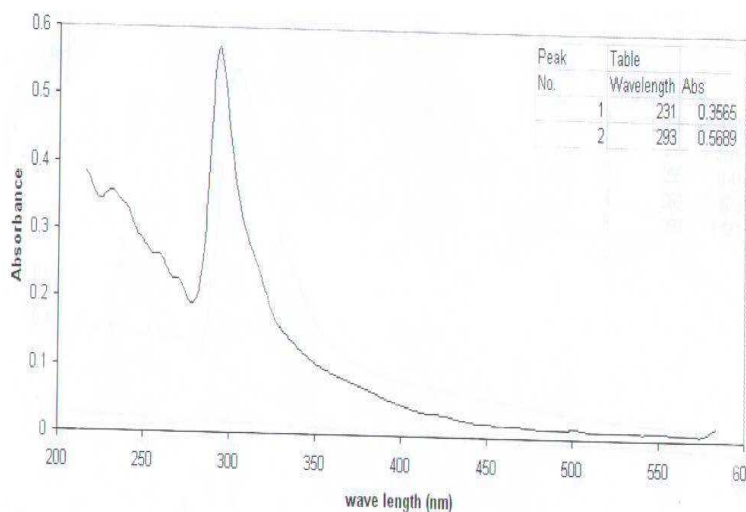
Figure(3):The infrared spectra of the complexes [II]



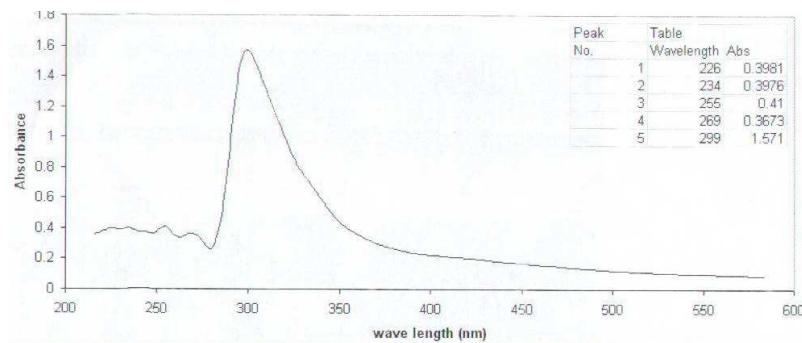
Figure(4):The infrared spectra of the complexes [III]



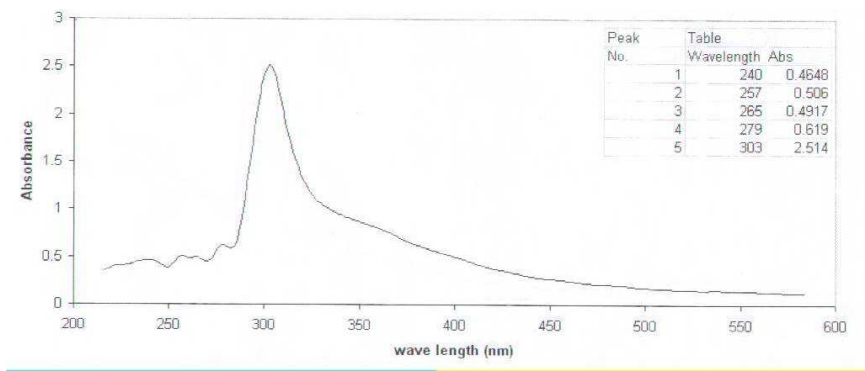
Figure(5):The infrared spectra of the complexes [IV]



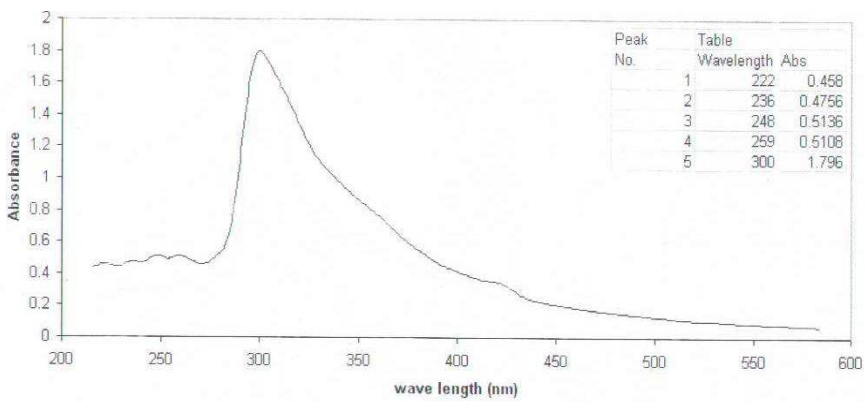
Figure(6):The U.V-visible spectra of the complexes [I]



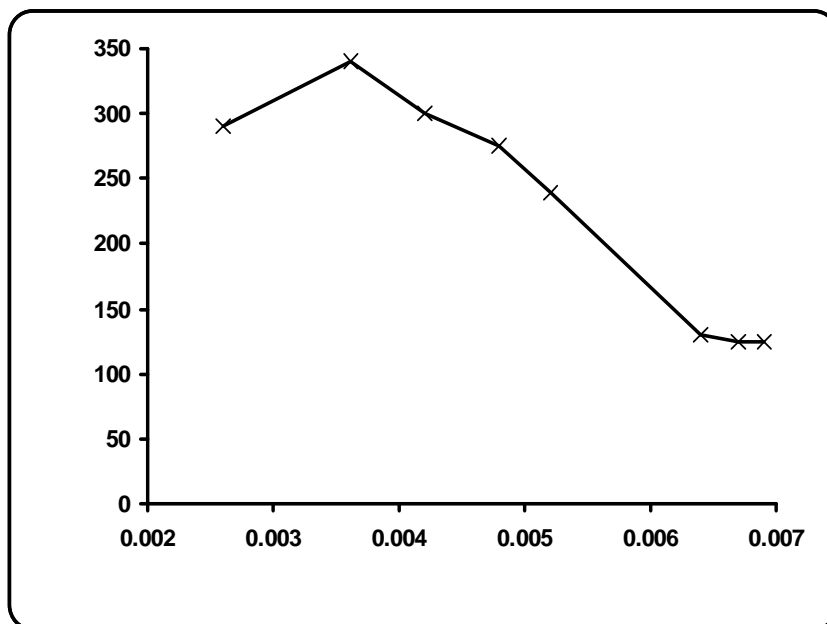
Figure(7):The U.V-visible spectra of the complexes [II]



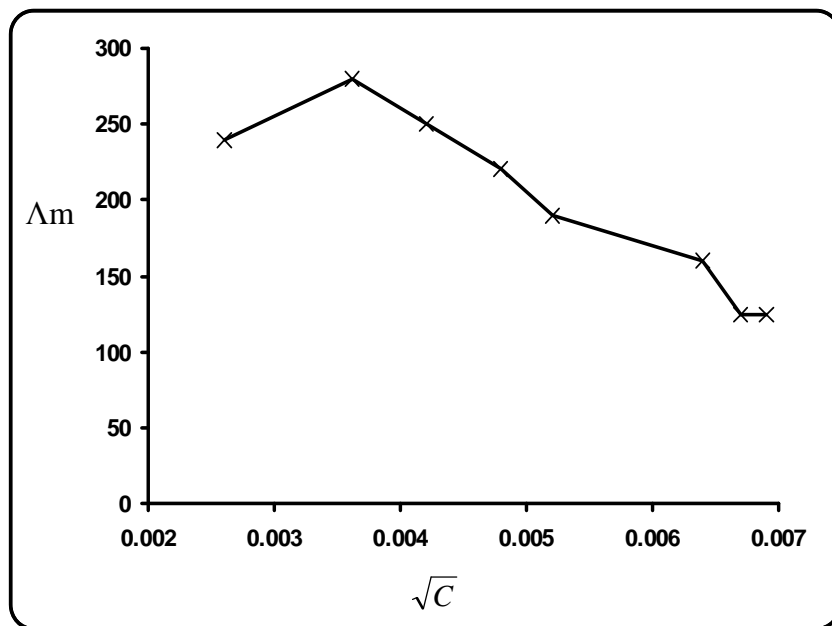
Figure(8):The U.V-visible spectra of the complexes [III]



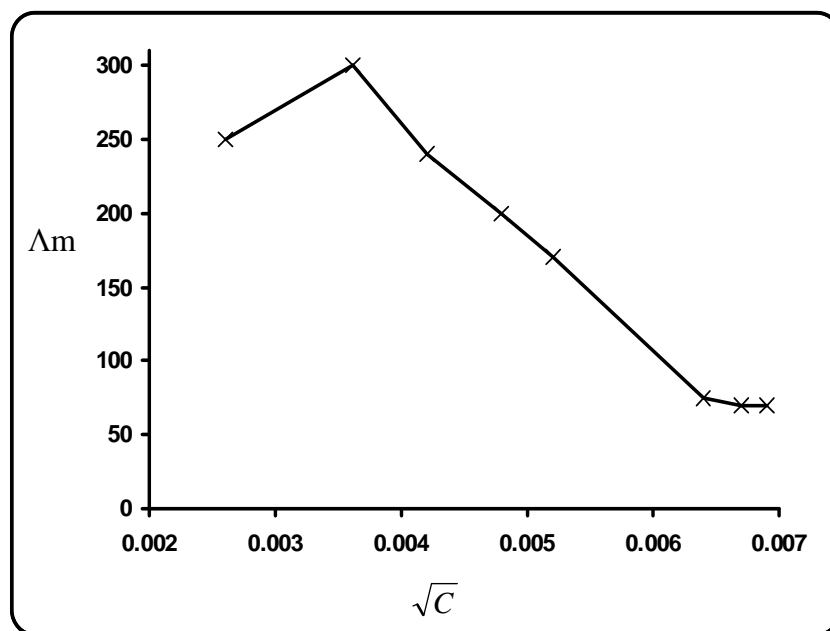
Figure(9):The U.V-visible spectra of the complexes [IV]



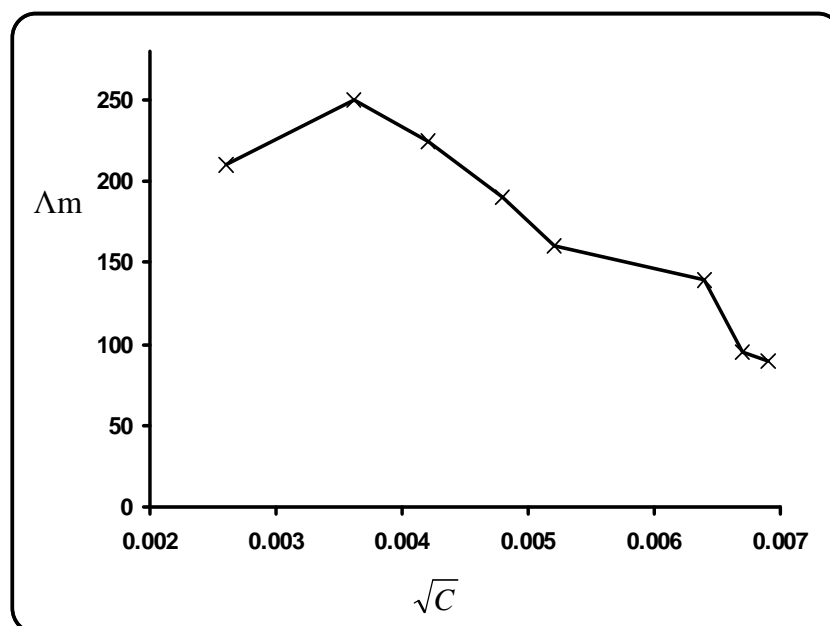
Figure(10): the molar conductivity vs square root of concentration of the complex(I)



Figure(11): the molar conductivity vs square root of concentration of the complex(II)



Figure(12): the molar conductivity vs square root of concentration of the complex(III)



Figure(13): the molar conductivity vs square root of concentration of the complex(IV)

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الخلاصة:

حضرة اربع مركبات من نوع [M(AA)(CBDCA)] حيث M = pt , pd ; AA= histidine (His) , tryptophan (Trp) ; CBDCA=1,1-cyclo-butandicarboxylate). وقد شخّصت هذه المركبات باستخدام اطياف الاشعة تحت الحمراء (IR) , الاشعة المرئية وفوق البنفسجية (UV-Visible) والتوصيلية المولارية . وقد اثبتت الدراسة ان الاحماض الامينية ترتبط بايون البلاتين الثنائي والبلاديوم الثنائي بنمط ثنائي . واثبتت اطياف الاشعة المرئية وفوق البنفسجية بان المركبات هي مربع مستوي وتمتلك مجموعة نقطية هي C_{2V} وظهرت المركبات سلوك الكتروليت ضعيف في مذيب DMF.