Synthesis and characterization of complexes with some amino acids

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Abstract :

This project includes and in deification the complexes of amino acid L-serine (serH) L-cysteine (cysH) , and L-threonin ($thrH_2$) with some ions of trace elements such as K_2ptCl_6 ,

$Cu(NO_3)_2.3H_2O$, $ZnCl_2$ and $HgCl_2$ were studied by Infrared-spectra ‘NMR from this identification studies can give the structure formed for the complexes with were prepare with amino acids :

$Na[pt(ser)Cl_4]$ $.0.5C_2H_5OH$,$[Hg(ser)_2]$ $.9H_2O$, $[Cu(ser)_2]$, $Na[pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$ and

Keywords: metal complexes – Amino acids – Characterization .

تحضير وتشخيص بعض من معقدات العناصر الانتقالية مع بعض من الأحماض الأمينية

المستخلص

تم في هذا البحث تحضير وتشخيص بعض المعقدات من تفاعل بعض من ايونات العناصر هي: Z(II) , Cu(II) , Pt(IV) , Hg(II) مع الأحماض الأمينية مثل السستايين والسيرين والثرونين والتي شخصت بعدة طرق منها طيف الأشعة تحت الحمراء والمرئية وفوق البنفسجية بالإضافة الى استعمال الطرق الفيزيائية الأتية : درجة الانصهار والذائيبية والتوصيل الكهربائي لمحاليلها .

الكلمات المفتاحية: الأحماض الأمينية – المعقدات – التشخيص.

1.Introduction:

Amino acids are very necessary in biological bodies, and complex readily with the first transition series ions such as Cu(II) , Zn(II) , Hg(II) , and pt(IV) ion .There is a wealth of information on the structures transition metal complexes with amino acids . in the sixties , there were extensive studies on the reactions of metal ions with amino acids . However, most of these reactions were in solutions. For example ,

The four amino acid peptide containing the two proline in the middle and two cysteine (cys) in the terminal positions was synthesized and its mercury (II) complex was prepared [pires , *etal ;* 2012] The spectroscopic measurements such as UV- visible , cicular dichroism and Raman indicated that the binding of Hg(II) is to the two cysteine molecules forming the dithiolatemercury (II) complex .

In the same year , the Schiff-base pyrrai-L- histidine and its Co(II) , Ni(II) , Cu (II) and Zn complexes were synthesized and characterized using various techniques. These data showed that ligand is tridentate and the binding sites are azomethine nitrogen , imidazole nitrogen and oxygen of the carboxylate group. [Arish and Nair ;2012]

[Faliah , *etal ;* 2013] The reaction of chloride –salt of transition metal with sodium of the amino acid. the complexes have been characterized by means infrared spectra and atomic absorption analysis . the IR spectra and atomic absorption analysis show that amino acids acta bidentate ligands.

Amino acid – metal complexes were prepared from four different salts of copper and amino acids (L-Asparginine , L- Histidine , L-lysine) as ligand . The complexes were synthesize and characterized by elemental chemical analysis , electronic and infrared spectra IR spectroscopy confirms the ligand coordination to the metal ions through carboxyl and amino groups . [ptripthi and Aarti kamal;2015].

The synthesis and structural characterization of Co(II) complexes of amino acid Schiff bases was prepared from salicylaldehyde and Three amino acid (valine , leucine and Isoleucine) in basic medium . The metal complexes was synthesized by treating an ethanolic solution of the ligand with appropriate amount of metal salts [1:2] [M:L] ratio , The synthesized Schiff bass by ^{1}H NMR , MS ,molar conductance and magnatic measurements. [Mobrouk ,*etal* ; 2017].

[Maher, *etal* ; 2018] The reaction of urea and glycine with the metal ions Co(II) , Ni(II) and Cu(II) in ethanolic solution of $1M:1L_1 : 1L_2$ molar ratio .Led to the preparation of complexes of the general formula $[M(ur)(gly)(H_2O)_2]Cl$. elemental microanalysis , molar conductivity measurements , IR , ^1H NMR , and UV-vis spectroscopic were used for the characterization of the compound .

[Andriana , *etal* ; 2019] Two copper (II) complexes with ligands derived from β-amino acids, 2-(1-amino cyclohexyl) acetic acid L1 and 2-(amino -4-tert-butyl) cyclohexyl) acetic acid L2 , were synthesized and characterized by microanalysis , IR , UV- visble and EPR spectra . The spectroscopically predicted structure of the square-planer copper (II) complex with 2-(1-amino cyclohexeyl) acetic acid C1 was confirmed by single – crystal –ray analysis .the biological (antitumor activities and interaction with DNA) of the compounds were also investigated .

[Sikumarin 2021] This work a new series of Schiff base has been produced by the condensation of salicyldahyde and amino acid namely alanine . Thus derived ligand and its relative copper metal complex were characterized by UV-visble , IR and NMR analysis .based on the results of spectral analysis the structures of the ligand and complex were deduced the purity of the products were examined through . Thin layer chromatography (TLC) . The antimicrobial properties of both the ligands and its corresponding copper complex has deduced against human pathogens namely E.Coli Bacillus subtilis , Candida Albicans A satisfactory inhibition caused by the copper complex over the ligand is verified and reported .

Rasha Khider 2022] Synthesized by reaction of the diazonium salt of transe - 4 –amino – methylcyclohexane carboxylic acid with -3- amino phenol . the azo ligand was characterized by micro elemental analysis and TGA as well as spectroscopic techniques UV-vis , IR , H^1_ NMR , and LC_ Mass as well as conductivity and magnatic susceptibility were used to characterized the complexes of Ni(II) , pd(II) , and pt(IV) the following general formula has been given for the producted compounds based on the results obtained , wich are as follouing : the Ni(II) complex has a 1:1 metal – ligand ratio , but the pd(II) and pt(IV) complexes have a 1:2 metal – ligand ratio.

It is obvious from the above review that not much work has been done on the preparation of solid complexes of amino acids with transition metal ions such as Ni(II), Co(II), Cu(II), Zn(II), Hg(II), Cd(II) and Pt(IV). Most of the work that has been reported was in solutions.

Therefore, the aim of this work has been to prepare complexes of the available amino acids with various salts of transition and non-transition metal ions such as Zn(II), Hg(II), Cu(II), Cd(II), Ni(II), Co(II) and Pt(IV) and to characterize them using all the available physical techniques such as conductivity measurements, IR, UV-visible and NMR spectral analysis wherever possible. We hope that some of them might have biological activity.

2. EXPERIMENTAL

2.1 Materials

The solvents used were analytical reagent grade and were used as purchased unless otherwise mentioned.

Potassium bromide (KBr), L-serine ($C_3H_7O_3N$), copper(II) nitrate trihydrate [$Cu(NO_3)_2.3H_2O$] and potassium hexachloroplatinate (IV) [K_2PtCl_6] were purchased from BDH, Poole, United Kingdom.

L-Cysteine ($C_3H_7O_2NS$) and L-phenylalanine ($C_9H_{11}O_2N$) were purchased from Park, United Kingdom.

Zinc(II) chloride ($ZnCl_2$) and sodium hydrogen carbonate ($NaHCO_3$) were purchased from S.D. Fine-Chem LTd, Mumbai.

Mercury(II) chloride ($HgCl_2$) was purchased from GCC, U.K. L-Threonine ($C_4H_9O_3N$) was purchased from Sigma,U.S.A.

2.2 Reactions of Metal Salts with Amino Acids

The metal salts[♣] of Cu(II), Zn(II), Hg(II) and Pt(IV) were reacted with the sodium salts of the following amino acids: L-phenylalanine (pheH), L-serine (serH), L-cysteine (cysH) and L-threonine (thrH_2). All of the isolated complexes were dried at 40°C. They were obtained by the following procedures A concentrated aqueous solution of the sodium salt of the phenylalanine [pheH 0.49 g , 3.0mmol] L-serine[serH 0.043g 0.41mmol] L-cysteine L-thronine [0.049g 0.41mmol] and $NaHCO_3$ (o.34g , o.41mmol) in deionized water (10ml) . The reaction mixture was refluxed at $70C^0$ for 5 hours and then filtered . The filtrate was evaporated to dryness under reduced pressure . The

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[♣] It is to be mentioned that other metal salts were used as shown in the Introduction. However, only the ions that gave good results are discussed here.

precipitate formed was washed well with ethanol , acetone and diethyl ether by centrifuge .

2.3 Physical Measurements

The elemental analyses for C, H, N, and S for the isolated complexes were run by the Jordan University of Science and Technology Laboratories, Jordan and Nanyang Technological University, Singapore. Melting points were determined on an electrothermal melting point apparatus and were uncorrected.

The infrared absorption spectra were recorded on an FT-IR Tensor 27 spectrometer, Bruker spectrum 2000 over the range 4000-300 cm–1. Potassium bromide pellets were used.

Conductivity measurements were carried out at 25°C using 1×10–3 M solutions in deionized water on a BC 3020 digital conductivity meter.

Electronic absorption spectra were recorded on a double beam spectrometer "Shimadzu Corporation" UV-2401 (PC), using 1×10–5 M solutions in dimetyl sulfoxide at 25°C.

The ^{1}H- and ^{13}C-NMR spectra were recorded on a 400 and 100 MHz, respectively, Bruker Avanced III spectrometer . Deuterated dimethyl sulfoxide (DMSO-d6) was used as a solvent with tetramethylsilane (Me4Si) as an internal standard.

3. Results and Discussion

The sodium salts of the amino acids were prepared by mixing 1:1 molar ratios of the amino acids and $NaHCO_3$ in water. The complexes were synthesized by using metal: ligand molar ratios of 1:1 or 1:2 with slight excess of the ligand in some cases. The reaction mixtures were either stirred at room temperature for about 3 hours until precipitates appeared, as in the case of the reactions of Hg(II), Zn(II), Cu(II) salts with phenylalaninate, Hg(II) with serinate and Pt(IV) with cysteinate, or the mixtures were refluxed at about 70^{o}C for about 5-7 hours. In the case of the reaction of Cu(II) with alaninate, a blue solid was formed upon reflux while its reaction with serinate gave a clear blue solution which was allowed to cool then treated with excess acetone to give a blue solid. In the remaining reactions, the filtrates were evaporated to dryness under reduced pressure. The products formed in all cases were washed well with ethanol, acetone and diethyl ether by centrifuge and dried at 40°C.

The isolated complexes are all stable. The Pt(IV) complexes are yellow, the Cu(II) complexes are blue while both the Zn(II) and Hg(II) complexes are white solids. They have high decomposition points (186-250°C) and were obtained in almost good yields as shown in (Table 1). They are insoluble in common organic solvents (Table 2) such as ethanol and acetone. Moreover, they are all insoluble in DMSO and DMF except $N_3[Pt(thr)_2Cl_3].2H_2O$ soluble in DMSO .The complex $[Cu(phe)_2].2.5H_2O$ insoluble in common available solvents. Some complexes are soluble in water as shown in (Table 2). They were characterized by elemental analyses (Table 3).

Therefore, no conductivity measurements could be done for them. However, their insolubility might support their non-ionic nature. The complexes $[Hg(ser)_2].9H_2O$ and $[Cu(ser)_2]$ behave as neutral non-electrolytes in water. The slight conductance observed may be due to some dissociation, decomposition or hydrolysis of these complexes. The complex $Na[Pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$ showed a molar conductance of 91 ohm^{-1} cm^2 mol^{-1} which suggests that the complex is a 1:1 electrolyte. The complex $Na[pt(ser)Cl_4].0.5C_2H_5OH$

Undergoes hydrolysis in aqueous solution . on the other hand, the complex $Na_3[Pt (thr)_2Cl_3]. 2.H_2O$ gave a value of 437 ohm^{-1} cm^2 mpl^{-1} indicating the presence of 4 ions.

Table 1 : Some physical properties of the isolated complexes

Complex	Yield (%)	Color	Decomp. point (°C)
$[Cu(phe)_2].2.5 H_2O$	86	blue	250
$Na[Pt(ser)Cl_4].0.5C_2H_5OH$	57	yellow	176
$[Hg(ser)_2].9H_2O$	79	white	179
$[Cu(ser)_2]$	72	blue	200
$Na[Pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$	50	yellow	188
$Na_3[Pt(thr)_2Cl_3].2H_2O$	47	yellow	186

Table 2: Solubility of the complexes

Compound	EtOH	H_2O	Acetone	DMSO	DMF
$[Cu(phe)_2].2.5H_2O$	-	-	-	-	-
$Na[Pt(ser)Cl_4].0.5C_2H_5OH$	-	+	-	-	-
$[Hg(ser)_2].9H_2O$	-	+	-	-	-
$[Cu(ser)_2]$	-	+	-	-	-
$Na[Pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$	-	±	-	-	-
$Na_3[Pt(thr)_2Cl_3].2H_2O$	-	+	-	+	-

(+) Soluble, (±) Slightly soluble, (-) Insoluble

Table 3 : Elemental analysis of the complexes

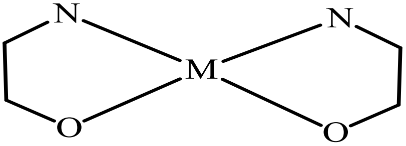
Compound	Analysis: calculated (found) %			
	C	H	N	S
$[Cu(phe)_2].2.5H_2O$	49.48 (49.488)	5.77 (4.888)	6.41 (6.408)	
$Na[Pt(ser)Cl_4].0.5C_2H_5 OH$	9.87 (9.929)	1.86 (1.929)	2.88 (2.967)	
$[Hg(ser)_2].9H_2O$	12.62 (12.21)	5.30 (5.53)	4.91 (5.44)	
$[Cu(ser)_2]$	26.521 (26.722)	4.451 (4.511)	10.311 (10.682)	
$Na[Pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$	13.098 (12.889)	3.298 (2.911)	4.365 (4.181)	9.991 (10.196)
$Na_3[Pt(thr)_2Cl_3].2H_2O$	14.996 (14.867)	2.831 (2.869)	4.373 (4.484)	

IR Spectra

The IR spectra of our complexes were studied along with their corresponding and related free amino acids. The main points are given below:

The presence of a medium broad band centered at 3222 cm^{-1}, which may be assigned to ν(N–H), and very strong broad band at 1623 cm^{-1}, which may be assigned to ν(COO^-), in the IR spectrum of $Na[Pt(ser)Cl_4].0.5C_2H_5OH$, confirms the bidentate coordination of the serinate anion through the $-NH_2$ and $-COO^-$ groups to Pt(IV). The strong broad band at 3418 cm^{-1} with a broad shoulder at 3500 cm^{-1} may be assigned to ν(O–H) of the hydroxyl group of serinate and ethanol. The low frequency region shows two bands at 545 and 532 cm^{-1} which may be due to ν(Pt–N) and ν(Pt–O). Unfortunately, the unavailability of CsI has hindered our studies since ν(M–Cl) appears at lower values. Potassium bromide is transparent up till 400 cm^{-1} only.

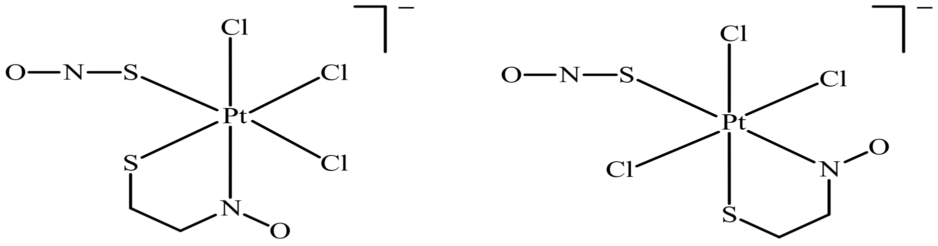
The IR spectra of the Hg(II) and Cu(II) complexes with serinate show bands that confirm the coordination of this anion to the metal ions through the $-NH_2$ and $-COO^-$ groups. The IR spectrum of $[Hg(ser)_2].9H_2O$ shows a very strong band centered at 3233 cm^{-1}, which is assigned to ν(N–H) and a very strong broad band centered at 1596 cm^{-1}, which may be assigned to $\nu(COO^-)$ with a large contribution of $\delta(NH_2)$. However, the IR of complex $[Cu(ser)_2]$ shows the ν(N–H) as a very strong very broad band in the range 3345-3132 cm^{-1} with a very strong band at 1666 $cm^{-1,}$ which may be assigned to $\nu(COO^-)$. The Hg(II) complex has a very strong, very broad band at 3468 cm^{-1}, which is assigned to ν(O–H) of the hydroxyl group of serinate and of water of hydration. The Cu(II) shows the ν(O–H) of the hydroxyl group of serinate at 3425cm^{-1}. Both complexes have four bands in the low-frequency region . which are not present in the free ligand and that can be assigned to ν(M–N) and ν(M–O). This number suggests that both complexes have a cis- configuration as shown below:



NO = serinate; M = Hg(II), Cu(II)

cis- configuration

As we mentioned before, the Hg(II) complex should be tetrahedral, since it has a d^{10} configuration with sp^3 hybridization, whereas the Cu(II), which has a d^9 configuration, can adopt a square planar or tetrahedral geometry but with some distortion due to Jahn-Teller effect [Miesslar *etal* ;2011]. Cysteine has three potential coordination sites: S, N and O. The IR spectra have been used to determine its mode of bonding [Nakamoto ; 2003]. The IR spectrum of free cysteine has a medium broad band centered at 3449 cm^{-1} and another very strong broad one centered at 3029 cm^{-1} which are assigned to ν(N–H). The corresponding bands in the complex Na $[Pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$ appear as very strong bands at 3202 and 3102 $cm^{-1.}$ Moreover, the spectrum also has a very strong broad band at 3433 cm^{-1}, which may be assigned to ν(O–H) of water of hydration and ethanol. Its high intensity indicates also that it could be in addition, due to ν(N–H) of free $–NH_2$ groups, i.e., one of the anions has uncoordinated $–NH_2$ group. Furthermore, the band which appears in the IR spectrum of the free ligand at 2586 cm^{-1}, that is assigned to ν(S–H), is absent from the spectrum of the complex. The $\nu(COO^-)$ which appears as a very strong band at 1723 cm^{-1} confirms that the carboxyl group is not ionized, i.e., it exists as –COOH . These results suggest that the cysteinate ion (cysH) is bonded through the $–S^-$ ion and an $–NH_2$ group while the carboxyl group is free and protonated. Cysteine has been reported to coordinate to metal ions such as palladium and platinum through the $–S^-$ ion and $–NH_2$ group [Elsherif *etal*; 2011] . The low-frequency region shows four bands at 516 (split), 478 and 439 cm^{-1}, which may be assigned to ν(Pt–N) and ν(Pt–S). Unfortunately, the unavailability of CsI makes it not possible to assign the ν(Pt–Cl) and determine whether the complex has a mer- or fac- configuration, as shown below:



fac-isomer **mer-isomer**

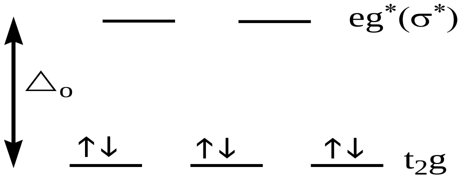
ONS = cysteinate

The IR spectrum of $Na_3[Pt(thr)_2Cl_3].2H_2O$ is completely different from that of free threonine . It shows a very strong and very broad band centered at 3425 cm^{-1}, which may be assigned to ν(O–H) of water of hydration and to ν(N–H) . Its very high intensity suggest that there is extensive H-bonding in the complex. The very strong broad peak centered at 3260 is assigned to ν(N–H) of bonded $–NH_2$ groups. However, the presence of two very strong bands in the region of $\nu(COO^-)$ at 1641 and 1623 cm^{-1} suggest that there are two types of carboxylate groups, bonded and free, respectively. Free threonine shows the corresponding band at 1628 cm^{-1}. Moreover, we mentioned earlier that conductivity measurements support the proposed formula and indicate that the complex behaves as a 1:3 electrolyte in water . This means that one of the carboxylate groups is free and not protonated and that all the hydroxyl groups of threoninate are ionized. The NMR measurements will support this conclusion later. The low-frequency region shows, the following bands: 558, 498, 450 and 423 cm^{-1}. However, free threonine shows similar bands at somehow close frequencies at 562, 491, 447 and 419 cm^{-1}. Thus these bands are not definitely for ν(Pt–N) and ν(Pt–O).

Electronic Spectra

The electronic absorption spectra of our Pt(IV) complexes were measured in DMSO. The spectra of $[Cu(ser)_2]$ and $[Hg(ser)_2].9H_2O$ were measured in H_2O while for $[Cu(phe)_2].2.5H_2O$ no spectra could be measured due to their insufficient solubility in common organic solvents. The bands were assigned by comparing them with reported data for complexes of amino acids and their derivatives [Elsherif *etal*; 2011].

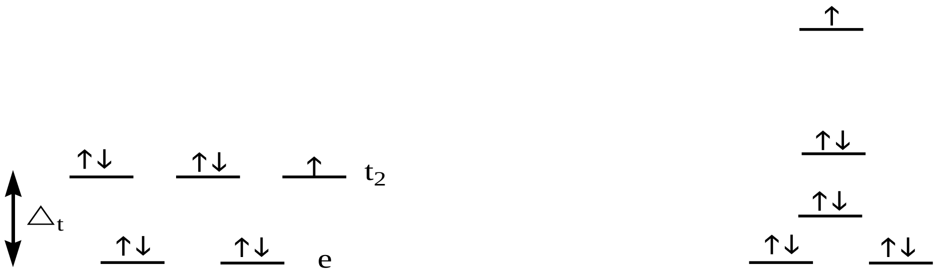
However, all complexes of Pt(IV) with d^6 configuration are expected to be octahedral, low spin and diamagnetic as shown below:



d-orbital splitting diagram for Pt(IV)

The spectra of Pt(IV) complexes with cysteinate are characterized by the presence of an LMCT band, possibly from the chloride ions to eg*, in the range 254-259 nm while the alaninate complex shows other CT bands at 265 and 272 nm. However, the complexes with phenylalaninate and threoninate show similar broad LMCT bands at 339 and 341 nm which might indicate that both are from the same ligand which is possibly the bonded chloride to eg* level.

All these bands in addition to the d-d transitions, are expected to give the Pt(IV) complexes their characteristic yellow color . On the other hand, the Cu(II) ion with a d^9 configuration and a coordination number of four might have tetrahedral or square planar structures but distorted due to Jahn-Teller effect [Cotton *etal;*1999] . Both structures are paramagnetic as shown below:

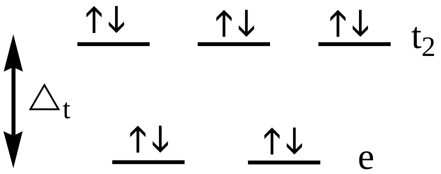


Tetrahedral Square planar

d-orbital splitting diagrams for Cu(II)

Moreover, both can accept electrons from the ligand giving rise to LMCT bands and have d-d transitions. All the Cu(II) complexes, prepared in this study, have a blue color which is characteristic of this ion. However, only LMCT bands are observed.

Furthermore, since Zn(II) and Hg(II) have d^{10} configuration, then their structures should be tetrahedral with no d-d transitions since all d orbitals are filled as shown below:



Tetrahedral

d-orbital splitting diagrams for Zn(II) and Hg(II)

NMR Spectra

The spectra of some of our complexes namely: $Na[Pt(ser)Cl_4].0.5C_2H_5OH$ and $Na_3[Pt(thr)_2Cl_3].2H_2O$ were carried out in deuterated D_2O and DMSO-$d_{6,}$ respectively, while D_2O was used for the free amino acids. The insufficient solubility of the other isolated complexes in the available solvents hindered their NMR studies.

Furthermore, it has been reported that the coordination of amino acids to metal ions, like Pd(II) and Pt(II), also produces downfield shifts for the ligand carbon signals especially those of the carboxylate group (COO^-) and α–carbons. The extent of downfield shifts for COO^- and α-C are 5-12 and 3-6 ppm, respectively. Such values can be clearly used to confirm the bonding mode of amino acids through the $–NH_2$ and $–COO^-$ groups [Krylova and Matveeva 2006] the latter reference uses also X-ray structure to confirm this mode of bonding .

Therefore, in the present study our main concern will be on ^{13}C-NMR spectra. So, we measured the ^{13}C-NMR spectra in D_2O for the free amino acids: L-phenylalanine, L-serine and L-threonine. The signals corresponding to the C of the carboxyl groups are: 173.88, 172.31 and 172.74 ppm, respectively. The signals corresponding to the α-C of these amino acids are: 55.99, 56.32 and 60.33 ppm, respectively.

4. CONCLUSION

In the present study, we managed to prepare some complexes of amino acids with Pt(IV), Cu(II), Hg(II) and Zn(II). The reactions of metal salts of these ions namely, K_2PtCl_6 ,$Cu(NO_3)_2.3H_2O$,$HgCl_2$ and $ZnCl_2$ with amino acid anions resulted in the formation of the following types of complexes :

i. Mono-chelated complexes namely: $Na[Pt(ser)Cl_4].0.5C_2H_5OH$.

ii. Bis-chelated complexes namely: $[Cu(ser)_2]$, $[Hg(ser)_2].9H_2O$, ,$[Cu(phe)_2].2.5H_2O$. $Na[pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$ and $Na_3[pt(thr)_2Cl_3].2H_2O$.

The amino acid anions are found to coordinate as bidentate ligands through both $–NH_2$ and $–COO^-$ groups .However , the cysteinate complex $Na[pt(cys)_2Cl_3].0.5C_2H_5OH.3H_2O$ is bonded through the $–NH_2$ group and $–S^-$ ion .

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